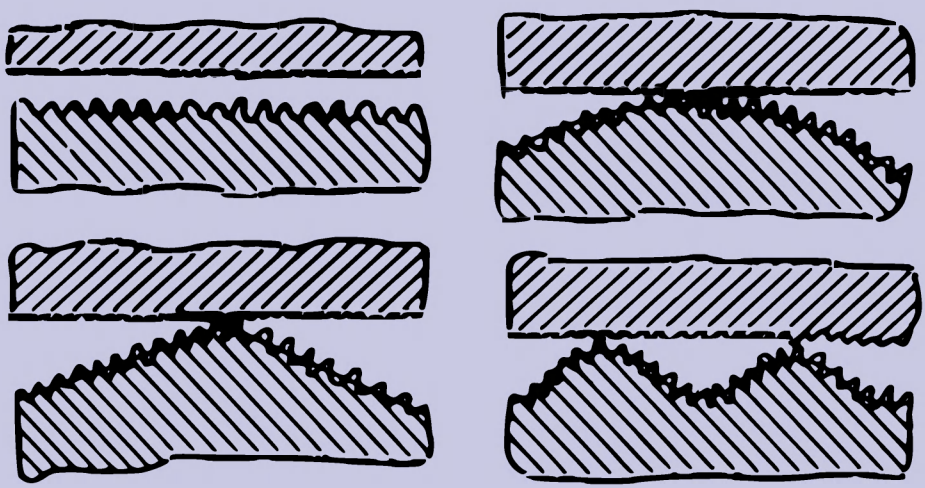
N.F. Kazakov (Ed.)

DIFFUSION BONDINGOF MATERIALS



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Diffusion Bonding of Materials

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Preface

The widely used resistance and fusion welding processes cannot overcome the incompatibility barrier inherent in metals, alloys, and nonmetals. The barrier has been cleared with vacuum diffusion bonding (Inventor's Certificate No. 112460, USSR).

Diffusion bonding in vacuum does not require any expensive solders, electrodes, fluxes, or shielding gases. A weldment needs no subsequent machining as the process leaves no scale, slag, or flash; nor is there any associated loss of metal. A diffusion-bonded weldment does not gain in weight, as inevitably happens in fusion welding, brazing, or cementing. There is no warpage to guard against, and the properties of the materials in the joint area remain unaffected. The process is conducted at moderate temperatures and pressures, so the products are of a higher quality and have a longer service life.

Diffusion bonding is a process of high productivity, readily amenable to automatic control. It is capable, in a single operation, of joining hundreds or even thousands of workpieces produced by stamping, rolling or extrusion into complex shapes or structures, with the materials utilized to a high degree of completion. This has all made it possible to combine in diffusion bonding two distinct operations, fit-up and joining proper.

Joints made by diffusion bonding meet the requirements for most critical structures in terms of strength, toughness, tightness, and resistance to heat and corrosion. As the process is conducted under a vacuum, a diffusion-bonded joint has a minimum of impurity content, even in the case of highly reactive metals. As compared with shielding gases, vacuum does not involve any transportation and storage expenses.

Diffusion bonding can join over 730 pairs of dissimilar metals. In the USSR, over 960 vacuum diffusion bonding units are in operation. The economic return from their use has run into about a 100 million roubles in recent years.

Outside the Soviet Union, the process has been granted patents in Britain, Belgium, the GDR, the United States, France, the FRG, Sweden, and Japan.

Several organizations in the Soviet Union (the Fundamental Research Laboratory for Vacuum Diffusion Bonding and others) have been the first to develop advanced modifications of diffusion bonding applicable to solid inorganic materials, vacuum diffusion bonding machines using various sources of heat and pressing systems, and also automatically controlled units which have substantially expanded the field of application for the process.

This book is an attempt to pool the experience in vacuum diffusion bonding accumulated by a number of mechanical engineering works, research establishments, and colleges.

The Authors

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An Outline of Diffusion Bonding in Vacuum

N. F. Kazakov

It is customary to class the existing welding processes into fusion welding and pressure welding. Accordingly, the definition adopted in the Soviet Union describes welding "as a process to make permanent joints by establishing interatomic bonds between workpieces through the action of local or general heating or plastic deformation, or both."

For interatomic bonds to be established, the workpieces must be brought together within the atomic spacing. Unfortunately, the surface geometry, chemical conditions, and physical state would usually stand in the way of bringing the pieces within the interatomic distance. Real surfaces are never ideally smooth or clean. Even carefully machined, a surface will show microscopic asperities with a height of hundreds or even thousands of molecular layers. So when two pieces are brought together, they will only come in contact at asperities. Also, the surface of any solid holds atoms from the surrounding medium, above all oxygen atoms, which form strong chemical bonds with the surface atoms. The simplest way to remove all of these obstacles is offered by fusion welding.

In *fusion welding*, the pieces to be joined are melted at their edges, the adsorbed substances vaporize or melt, and closure to within the interatomic distance takes place spontaneously as a weld pool is built up. A permanent joint forms as the weld pool cools and coalesces into a single body (along with the molten filler metal, if any has been used).

In *pressure welding*, the pieces to be joined are brought to within the atomic spacing through plastic deformation in the weld zone. Sometimes the mating surfaces are cleaned by, say, heating in vacuum or in a reducing atmosphere, autovacuum cleaning, etc. A full-strength joint is made owing to a physical contact, chemical bonds (which may be metallic, ionic, covalent, or their combination) between the atoms on the surfaces of the workpieces, and volumetric transformations.

The field of pressure welding can be divided into several subdivisions, such as resistance welding, gas-pressure welding, diffusion bonding, friction welding, ultrasonic bonding, stored-energy welding, explosion welding, etc.

Diffusion bonding, as its name implies, involves the interdiffusion of atoms across the interface of the weld in the solid and, sometimes, the liquid state (when a molten interlayer is used). The applied bonding pressure (or pressing load) causes the edges to move within the range of atomic forces. If there is a molten interlayer, the decisive factor is the pressing load which expells the interlayer from the joint. The pressing load is raised until all of the interlayer is squeezed out.

Diffusion bonding is carried out in suitably designed units fairly simple in their arrangement (Fig. 1-1). The pieces to be joined, *2*, are loaded into a vacuum chamber, *1* (which is cooled by running water in the course of welding). A vacuum system, *3*, maintains in the chamber sufficient rarefaction as a way of protecting the workpieces against intensive oxidation as they are heated for joining. Heat is supplied by the inductor, *4*, of a high-frequency oscillator, *5*. The pressing load applied to the workpieces is supplied by a hydraulic system, *6*. When the joint is completed, the weldment is brought down to the specified temperature in the vacuum chamber.

The success or failure of the process is decided by three variables which need a constant watch and careful adjustment. These variables are the bonding temperature, the bonding pressure (or pressing load), and the holding time (duration of pressure).

The *bonding temperature* should be anywhere between 50% and 70% of the melting point of the most fusible metal in the composition. Elevated temperature aids the interdiffusion of atoms across the interface of the weld, and this assists surface deformation (the crushing of surface asperities).

The *bonding pressure* or *pressing load* should ensure a tight contact between the edges of the pieces. It must be sufficient to aid deformation of surface asperities and to fill all the voids in the weld zone. If the pressure is not sufficient, some of the voids will be left unfilled, and the strength of the joint will be impaired. Importantly, the pressing load disperses oxide films — this leaves a clean surface, and aids the diffusion and coalescence.

The *holding time* (duration of pressure), at a specified bonding temperature and a specified bonding pressure, must, in most cases, be kept to a minimum from

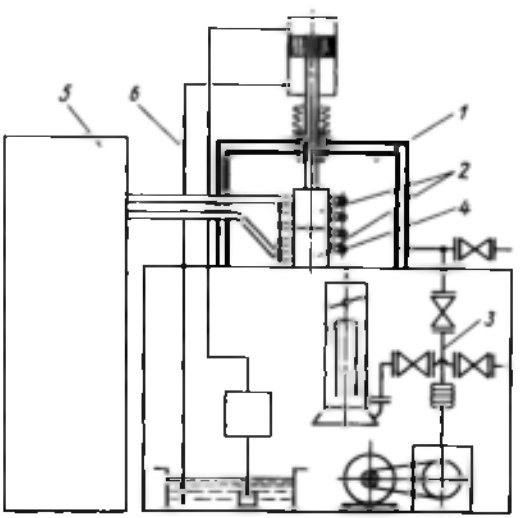


FIG. 1-1 Basic features of a vacuum diffusion bonding unit

physical and economic considerations. It should be just sufficient for an intimate contact to be formed and for diffusion processes to take place. An excessive diffusion time might leave voids in the weld zone or even change the chemical composition of the metal or lead to the formation of brittle intermetallic phases (when dissimilar metals or alloys are being joined).

The vacuum in the welding chamber is usually maintained at 10^{-1}-10^{-3} Pa. Sometimes, the medium in the chamber may be an inert gas (dry argon or dry helium with a dew point of below 216 K), hydrogen, or various gas mixtures.

For the joint to be sound, there must be a good surface contact, the edges must be smooth and fit to each other snugly. Soft metals, such as aluminium, magnesium, tin, and gold, need to be surface-finished prior to diffusion bonding so that the surface asperities are not more than 40 to 80 μm high. Hard alloys, refractory metals, high-temperature alloys, tool steels, and nonmetal materials call for a more thorough surface finish. Surface asperities (roughness) in such cases should be not more than 2.5 μm high or even lower. The use of an interlayer of a readily deformable material is desirable.

To repeat, diffusion bonding is a process by which a joint can be made between similar and dissimilar metals, alloys, and nonmetals, through the action of diffusion of atoms across the interface, brought about by the bonding pressure and heat applied for a specified length of time.

Sometimes, diffusion bonding is referred to as pressure joining, autovacuum welding, thermo-compression welding, solid-state or solid-phase welding, etc. This may introduce an element of confusion into the understanding of the essence and mechanism of diffusion bonding. To avoid all this, a relevant USSR State Standard defines both the process and the associated terms. As far back as 1962, N. F. Kazakov, who is credited with the invention of diffusion bonding, described diffusion bonding in the following terms: "In this case, a monolithic joint is due to the formation of metallic bonds as a result of local plastic deformation at elevated temperature, closest approach of the surfaces, and interdiffusion in the surface layers of the metals brought in contact." More recently diffusion bonding has been used to join both metals and nonmetals to metals and nonmetals, so in addition to metallic bonds other types can appear in the weld zone. Accordingly, the International Welding Institute has adopted a modified definition proposed by Kazakov: "Diffusion bonding of materials in solid state is a process for making a monolithic joint through the formation of bonds at atomic level, as a result of closure of the mating surfaces due to local plastic deformation at elevated temperature which aids interdiffusion at the surface layers of the materials being joined."

Since the principal factor responsible for the formation of the joint is diffusion, it appears that the term "diffusion bonding" or "diffusion welding" is most relevant. It includes the two key words, diffusion and bonding, that is, the processes by which pieces are joined into a single whole. The bonding variables (temperature, load and time) vary according to the kind of materials to be joined, surface finish, and the expected service conditions.

Diffusion bonding offers a number of advantages over fusion welding and brazing. This is clearly seen from their comparison in Table 1-1.

TABLE 1-1 Comparison of Diffusion Bonding and Other Joining Processes

Particulars	Fusion welding	Diffusion bonding	Brazing
Contacting method	Autogenous fusion, autogenous fusion and pressure, pressure and autogenous fusion	Pressure (no fusion)	Contact fusion, contact fusion and pressure, pressure and contact fusion
Bonding	Cohesive	Adhesive, diffusion	Cohesive, adhesive
Heating	Local	Local, total	Local, to al
Temperature	Melting point of parent metal	0.5-0.7 of melting point of parent metal	Somewhat above melting point of solder
Surface preparation	Less exacting	Careful	Less exacting
Fit-up	Lenient	Precise	With capillary gap
Materials	Metals, alloys	Metals, alloys, nonmetals	Metals, alloys, nonmetals
Joint formation	Gradual	Simultaneous	Simultaneous, gradual
Edge preparation	Yes	No	Yes
Joining of dissimilar materials	Limited	Unlimited	Unlimited
Stepwise conduct of process	Limited	Unlimited	Unlimited
Susceptibility to solidification cracking	Strong	None	Weak
Porosity	Shrinkage, blowholes	None	Blowholes, shrinkage, diffusion
Overlapping with heat treatment	No	Unlimited	Limited
Warpage	Heavy	None	Light
Principal types of joint	Butt, lap	Flat (butt, lap, tapered plug in socket, between cylinders, spherical, curvilinear)	Butt, lap
Joining in hard-to-reach places	Limited	Unlimited	Limited
Product precision	Low	Fairly high	High
Disassembly of joint	No	No	Yes
Vibration survival	Low	Very high	High
Corrosion resistance	Satisfactory	Fairly high	Low
Strength	Close to that of parent metal	That of parent metal	That of solder
Air pollution and radiation emission	Yes	No	Yes

A very important distinction of diffusion bonding is the high quality of joints. It is the only process known to preserve the properties inherent in monolithic materials, in both metal-to-metal and nonmetal joints. With properly selected process variables (temperature, pressing load, and time), the material at and adjacent to the joint will have the same strength and plasticity as the bulk of the parent material(s). When the process is conducted in vacuum, the mating surfaces are not only protected against further contamination, such as oxidation, but are cleaned, because the oxides present dissociate, sublime, or dissolve and diffuse into the bulk of the material. This is why a diffusion weld is free from incomplete bonding, oxide inclusions, cold and hot cracks, voids, warpage, loss of alloying elements, etc. Since the edges are brought in intimate contact, there is no need for fluxes, electrodes, solders, filler materials, etc. Diffusion-bonded parts usually retain the original values of ultimate tensile strength, angle of bend, impact toughness, vacuum tightness, etc. In terms of strength, ductility, tightness, and corrosion resistance, diffusion-bonded joints meet the requirements for the most critical applications. Diffusion bonding has extended the service life, quality and reliability by a factor of 10 to 12 for a wide range of products, and has made it possible to develop fundamentally new structures, to simplify techniques and procedures, and to replace scarce or expensive materials. A major distinction of diffusion bonding, the high stability of mechanical characteristics, enables one to limit quality control to a random check in which just a few parts are examined for quality and compliance with specifications instead of a large sample or the entire lot. This is especially valuable because simple, inexpensive and reliable techniques of nondestructive testing which could be used in a welding or an assembly shop are nonexistent in some cases.

Diffusion bonding can turn out weldments extremely intricate in shape, but with joints having the same properties as the parent materials. With it, one can make shapes that could not be fabricated earlier or which would be too expensive to make by conventional processes. Dimensional tolerances for diffusion-bonded products are the same or even more stringent than those for machined parts. Since diffusion bonding makes unnecessary most of the fasteners, there is less tendency towards stress-induced corrosion which is a crucial matter for products with a long service life. The materials to be joined may vary in thickness from a few micrometres (foil) to several metres.

In recent years, a theory of diffusion bonding has been developed and important results obtained on the diffusion processes involved in the formation of a monolithic bond between solid inorganic materials of any kind without affecting their physical and mechanical properties. Now diffusion bonding can be used to join not only similar, but also dissimilar metals and alloys widely differing in thermo-physical characteristics; the process can be used to weld heavy thicknesses and any complicated shapes from porous, fibre and powdered materials; to join nonmetals (such as glass, semiconductors, ceramics, pyrosils, quartz, graphite, ferrites, cermets, and the like) to metals; and to repair or reclaim worn or broken components for various machinery.

Now diffusion bonding has a companion — diffusion metallurgy which is gaining application on an ever wider scale. A process has been developed for thermal

vacuum treatment which extends the service life and reliability of machines and tools. Research has led to a variety of multicomponent composites which combine the properties of metals, alloys, and nonmetals.

By its nature, diffusion bonding lends itself readily to automation. Automated diffusion bonding can join in a single pass tens or even hundreds of components, fabricated by stamping, rolling, casting, or extrusion, into complex assemblies and structures with a high utilization of materials.

The basic variables of diffusion bonding (temperature, pressing load, vacuum, and holding time) can readily be programmed. As a rule, equipment for diffusion bonding is either semiautomatic with a minimum of manual labour, or automatic, operating with practically no human intervention. The high degree of mechanization of diffusion bonding equipment, the high quality and the longer life of products and the high sanitary standards of the environments make diffusion bonding a far more attractive process than any other conventional welding processes.

A further advantage of diffusion bonding is that it can join any components, however complicated their shape or cross section may be. Among other things, it can butt-join pipes and bars, to butt- and lap-weld flat pieces, and to make a variety of joints — corner, plug lap, coach, tapered, spherical, involute, etc. The configuration of the workpiece having a flat surface, and its dimensions do not affect the quality of the diffusion bond; they can only control the choice of the heat source and pressing arrangement to transfer the bonding pressure to the workpiece.

Until quite recently, diffusion bonding was applicable to a limited range of shapes and cross-sectional areas at the point of contact. Also, the space within the vacuum chamber was not utilized fully. To circumvent the limitations, new diffusion bonding units have been developed, such as multichamber (the rotary type), continuous (the airlock type), and hopper-loaded. There are also units operating to a preprogrammed sequence, with vacuum chambers set up directly on the workpiece, etc. These chambers can accommodate workpieces up to 750 kN in weight, up to 50 m in length, and 58.8 mm in diameter, and the blanks are fed in on air cushions.

Duffusion bonding has been used to fabricate silver- or copper-clad chemical units 3 m high and 1.86 m in diameter, wear-resistant dies to punch stampings for the magnetic cores of electric motors, cutting and measuring tools, metal-ceramic pressure-seal feed-throughs, ferrite and cermet assemblies, the elastic elements of transducers, sandwich panels, fluidics modules, radial-turbine wheels, turbine blades, porous tubes for the chemical and gas industries, valves, pistons and cylinder liners for internal-combustion engines, and many other products. In the electronics industry diffusion bonding is used to make and assemble slow-wave structures for some electron tubes, to join nickel foil 3 μm thick to a substantial component, and aluminium foil 8 μm thick to a copper grid. Diffusion bonds are vacuum-tight, heat-resistant, and vibration-proof, with the diffusion-bonded products retaining their high accuracy, dimensions, and shape.

Diffusion bonding is a highly economical process. It does not involve any expensive solders, special grades of wire or electrodes, fluxes, and shielding gases. Moreover, there is no need for the subsequent machining, so there is no associated loss of valuable metal. A diffusion-bonded product does not gain in weight as hap-

pens with ordinarily welded, brazed, or glued components. The properties of the metal in the weld zone remain as they were, so postheat treatment is not mandatory. Pieces of diffusion bonding equipment can be built into machining or assembly flow lines, so no facilities are necessary to transfer components between machining and welding shops. The power demand for diffusion bonding is one-fourth to one-sixth of what is taken up by, say, resistance welding.

Further advantages of diffusion bonding are high sanitary standards of the working environments and freedom from ultraviolet radiation, harmful gas emission, metal spatter, and finely divided dust.

Diffusion bonding has contributed to advances in other fields. As Academician G. N. Flerov believes, the discovery of the 106th element in the Periodic Table has been expedited by the Vacuum Diffusion Bonding Fundamental Research Laboratory which was asked to devise ways and means for reliably joining the working substance to the electrode of a source operating in the plasma discharge. An important project has been carried out for Academician A. M. Prokhorov, a Nobel Prize winner, in connection with his work on powerful gas lasers.

A major limitation of the process is that a good deal of time has to be spent on pumping out the vacuum chamber, cleaning and fitting up the pieces to be joined. Currently, work is under way to avoid this, and it may be expected that an answer will be found before long.

The success or failure in using diffusion bonding commercially depends on the scale of production and the quality of the equipment used.

Recently, a change of attitude has taken place as regards diffusion bonding. From a process applicable to materials which can be joined by conventional welding or brazing with difficulty, if at all, it has grown to a process readily available to join both small and large workpieces. Today, it is highly competitive with existing fusion welding and brazing processes.

Diffusion bonding is a promising process which is bound to gain more and more in importance as the use of alloyed steels, special alloys, nonmetallic and composite materials is increased. In the author's opinion, its further advance will take a two-pronged road. One will be concerned with further perfection of its techniques and procedures, methods of heating, transfer of pressing load, mechanization and automation, nondestructive testing, and mechanized and automated equipment. The other will be related to getting a better insight into the mechanism of diffusion bonding and bringing out the yet-untapped potentialities of the process in its application to large and critical components for instruments, apparatus, and machines.

Biblioghraphy

1. KAZAKOV, N. F. *Diffusion Bonding of Materials.* Mashinostroyenie, Moscow, 1976 (in Russian).
2. KOCHERGUIN, K. A. *Pressure Welding.* Mashinostroyenie, Leningrad, 1972 (in Russian).
3. *Standardization of Unit Operations and Processes.* VNIINMASH, Moscow, 1973 (in Russian).

2

A Theory of Diffusion Bonding

N. F. Kazakov

2.1 The Mechanism of Bond Formation in Pressure Welding

Although a multitude of welding processes are currently used on a commercial scale, it is pressure welding that is leading the field.

Pressure welding refers to a process in which a joint is made at temperatures below the melting point of the metals being joined, without any solder used, and under a pressing load sufficient to bring about the necessary plastic deformation of the pieces.

Several hypotheses have been advanced to explain how a bond is formed in solid state. One is the *film hypothesis* due to S. B. Ainbinder *et al.* By this hypothesis, all metals and alloys possess an equal property to seize when clean surfaces are brought together within the range of interatomic forces. The observed differences in weldability among various metals are explained by the presence of surface films. The oxide films which prevent joining can be hard, brittle, viscous, or plastic. When the metals being joined are subjected to cold plastic deformation, the hard and brittle films are broken up to expose clean metal layers which, on being brought together within the range of interatomic forces, form a strong bond. If at least one of the two metals has a plastic oxide film on its surface, plastic deformation will cause the oxide film to spread along with the underlying layers, and no bond will be formed. In some patterns of deformation, however, oxide films play a minor role.

The *recrystallization hypothesis* puts emphasis on recrystallization as the principal factor in bond formation. By this hypothesis, deformation and the accompanying strain hardening, coupled with exposure to relatively high temperatures at the interface, cause the atoms in the lattices of the materials being joined to move to other sites so that there appear, at their boundaries, grains common to both pieces with the result that a bond is formed. The proponents of the hypothesis have not been able, however, to present any convincing proof. Among other things, a diffusion bond can be formed at a temperature as low as 123 K; the weld zone displays high hardness; the rate of deformation does not affect the strength of the resultant bond obtained by cold welding; X-ray diffractometry reveals no recrystallization in cold-bonded aluminium; and a strong bond is produced with the formation of metallides (compounds of metals with metals and other elements — oxides, hydrides, nitrides, carbides, etc.) at the interface.

According to the *energy hypothesis*, for a diffusion bond to form the atoms of the metals being joined should be raised to what may be called the energy threshold of seizure or adhesion. At this threshold, the orientation of atomic bonds is no longer a factor, metallic bonds come into being between the atoms at the surfaces, and the

interface between the two pieces disappears. The combined plastic deformation necessary for the onset of seizure decreases in proportion to the increase in the energy of the atoms in the metals. For example, heating or elastic deformation, which raise the energy of the atoms, can bring the metal closer to the condition necessary for seizure to take place. In the opinion of A. P. Semenov, the seizure of metals is a nondiffusion process. At elevated temperatures, however, a diffusion process is superimposed on the nondiffusion seizure within the contact zone. The energy hypothesis fails to explain which properties of the metals being joined are responsible for the degree of seizure.

By the *dislocation hypothesis*, due to J. Friedel, E. I. Astrov, and some others, the joint plastic deformation causes dislocations to move to the surface. In doing so, they break up the oxide films present and produce steps each an interatomic distance high. One body of opinion is that the emergence of dislocations at the contact surface reduces resistance to plastic deformation and aids in joining the metals. Another believes that the emergence of dislocations increases the number of high and low spots on the surface, and this promotes a greater plastic deformation at the contact surfaces than within the bulk of the metals. Thus, seizure, or bond formation, is an outcome of the plastic flow of the metal within the contact zone. The proponents of the dislocation theory argue that seizure may also be accompanied by diffusion processes, but there is no consensus on the mechanism of seizure.

The *electron hypothesis* has been advanced by G. V. Samsonov *et al.* In their opinion, the surface seizure results in the formation of stable electron configurations involving the atoms of the metals in contact. If the electron configurations in the two metals being joined have high statistical weights, their adhesion is considerably weakened [13]. It has been demonstrated that knowledge of the electron configurations of metals and elemental atoms in chemical compounds gives an insight into their weldability, wettability, diffusion processes, etc.

In accord with the *diffusion hypothesis* [7-11], the formation of a sound bond between the surfaces in contact depends on the interdiffusion of atoms into the bulk of the specimens. The surface atoms of a metal have free, unfilled bonds (vacancies) which capture any atom moving within the range of interatomic forces. A free atom has an excess energy as compared with the atoms in a condensed system, and its attachment is accompanied by release of energy. This spontaneous union can be observed within volumes of a homogeneous liquid. The picture is different in solids. Now a substantial amount of additional energy has to be put in, and elaborate techniques have to be used in order to bring the atoms to be joined closer together.

There are many more hypotheses advanced to date; we have only dwelt on the most plausible and commonly accepted.

Despite the seeming simplicity of the process, pressure welding involves a sequence of stages or steps: the surfaces to be joined are first brought in contact; the surface oxide and adsorbed films are then broken up; the surface layers are activated as a result of deformation; volumetric diffusion processes take place, recrystallization follows, etc. These stages, undoubtedly overlapping in actual practice, testify to the complexity of pressure welding, because of which the mechanism

of the process cannot, at times, be explained from a single point of view. Lack of experimental data about the effect of the various factors and parameters on bond formation in pressure welding also contributes to the existing disagreement on the nature and mechanism of bonding. Yet, a general idea has of late taken shape. Most investigators agree that pressure welds are produced as a result of the metallic bonds that form between the metals being joined. Ya. I. Frenkel views any metal specimen as a collection of positively charged ions located, ideally, at the lattice sites, and a large number of free electrons interacting with many ions in that specimen at a time. This interaction of the electron cloud with ions is responsible for the existence of the specimen as a single body. Given certain conditions, a similar interaction may take place between the surfaces of two metal specimens being joined. When they are brought together to within the range of interatomic forces, the interaction causes the atoms to form a bond which is as strong as it is within the bulk of the metal. In other words, the atoms of a metal specimen tend to unite with similar or any other atoms and molecules. In doing so, they may form complexes, however large, or volumes of metal.

If two specimens brought in contact had ideally smooth, clean and parallel surfaces, metallic bonds sufficient for a joint to form and to have the same strength as the parent metal would be established spontaneously, and no external energy input would be necessary. Ideal surfaces are nonexistent, however. The surfaces of real metal specimens are always covered by films of oxides and adsorbed grease and gases, which interfere with the formation of metallic bonds, and are never smooth. Any specimen, no matter how carefully it has been machined, shows a wavy and rough surface with a multitude of microscopic asperities. So the pieces being joined first touch each other at the ridges of these high spots, and the contact is of necessity a discontinuous one. In the unloaded state, the contact area at each asperity varies with surface quality and some other factors and ranges between a fraction of a square micrometre and thousands of square micrometres. Although the number of contacting asperities at any given instant may be large, the total actual area of contact, ΣA_{act}, is only a small fraction of the nominal area of contact, A_n, being from $0.01\,A_n$ to $0.000\,01A_n$. Obviously, with the total area of contact of such a size the resultant joint can have a strength substantially lower than that of the parent metal, and then only if the interaction due to metallic bonds between the mating surfaces takes place at all, that is, only if there appear initial sites of seizure (adhesion) and their local strength is sufficiently high. In fact, even with a large number of seizure sites, no welded joint may have formed yet. So, seizure (adhesion) and welding are not the same because they occur on different scales. A single seizure site may be treated as the initiation of a welded joint, and for the latter to form it must spread over all or the greater part of the nominal contact area. For two metal specimens, A and B to be joined, they must be brought together to within a distance sufficient for a strong bond between their atoms to form. The energy of interaction between the atoms will then be a minimum

$$E = U - TS \tag{2-1}$$

where E is the free energy for constant volume, v, U is the internal energy of the system, T is the temperature of the joint, and S is the entropy.

When brought to within such a distance, r, the atoms form a most stable system, because any increase or decrease in interatomic distance would increase the energy of atomic interaction (Fig. 2-1). The above scheme can only occur in exceptional cases, such as when two metal specimens with juvenile surfaces are brought together under an ultra-high vacuum (1.33×10^{-6} Pa).

Actually, the situation is far more complex. As already noted, a real specimen, no matter how well it is machined, presents a microscopically rough surface. In the best of surface finishes, the asperities are 0.3 to 1 μm high. When two metal surfaces are brought together, the interatomic forces of attraction begin to be felt at distances anywhere between 10^{-4} and 5×10^{-4} μm. Therefore, if the pressing load is small and does not bring about marked plastic deformation, atomic interaction will be solely observed at individual asperities. In the clearances, only adhesion bonds between the metal and the gas or liquid molecules adsorbed on the metal surface will exist. For seizure to spread over a larger area and, as a consequence, for the actual welding to take place, it is necessary to apply either a high pressure which would bring about plastic deformation in the metal around the contact area, or a strong heat which would enhance the activity and mobility of the species in the crystal lattice. When both pressure and heat are applied together, they produce in the weld zone a concentration of energy which, in Academician P. A. Rebinder's words, is sufficient to bring about a rearrangement of the surface layers in the mating specimens, and also to give rise to the slower secondary processes, such as interdiffusion, recrystallization and so on. The latter can proceed either spontaneously or need far less energy than is necessary to form areas of direct contact between the specimens.

If, now, hot pressure welding is conducted in a vacuum, this will not only protect the metal surfaces against further contamination (such as oxidation), but also clean them through the desorption, sublimation, or diffusion of the oxide coating into the bulk of the material. This can produce a metallic bond at the contact surfaces, but this bond is sometimes not sufficiently strong to assure a good weld. A far better weld is made when the weld zone is extended into the bulk of the material. This extension occurs through interdiffusion which is a form of mass transfer.

The effect of diffusion processes on recrystallization and the formation of an intermediate zone varies with the welding temperature. If the two pieces being joined widely differ in physical and chemical properties, this zone may display changes in chemical bonds and composition. Within it, the type and parameters of the lattice and some physical properties of the parent metals may change gradually (from the

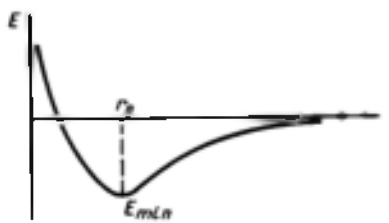


FIG. 2-1 Potential energy as a function of atomic spacing

properties associated with one piece to those of the other). In other words, pressure welding can produce a monolithic joint only if bonds are formed at the atomic level as the pieces to be joined are brought together and subjected to plastic deformation. The joint thus made gains in strength and reliability as interdiffusion promoted by heating causes the weld zone to expand.

2.2 The Basic Pattern of Diffusion Processes in Materials

Diffusion refers to the actual transport of mass in the form of discrete atoms through the lattice of a crystalline solid. Diffusion processes are fundamental in a large class of effects in metals and alloys (grain growth, polymorphism, relaxation, recrystallization, homogenizing heat treatment, precipitation hardening, surface impregnation, sintering, pressure welding, etc.) [3, 4, 5, 7-10].

As the present-day views hold, metals and alloys, when solid, are each a system of positive ions regularly distributed in space, and relatively free electrons which move within well-defined energy bands. The ions oscillate at a very high frequency (about 10^{13} Hz), and it is thermodynamically feasible for them to move to another, energetically more attractive site. This motion is greatly enhanced by a rise in temperature.

The diffusion (both self- and heterodiffusion) of atoms can take place only if the diffusing atoms have a sufficient store of energy for their migration through the lattice. At any temperature, the mean energy of oscillation of atoms in the lattice is a fixed quantity. This energy, however, varies from atom to atom in accord with laws of probability. Each atom in a state of continuous thermal vibration may collide with adjacent atoms. Through collisions, atoms pick up abnormally high velocities each corresponding to a particular local limit of temperature. In this way, the distribution of thermal energy within the lattice is subject to fluctuations. The distribution of atoms in the lattice by energy is shown in Fig. 2-2. The initial portion of the curve corresponds to the higher energy, E_1, displayed by a minority of atoms at temperature T_1, as compared with the mean energy, E_2, of the majority of atoms.

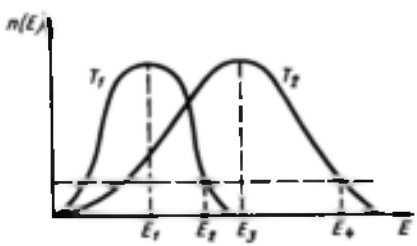


FIG. 2-2 Energy distribution of atoms in a metallic lattice

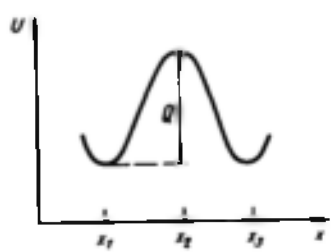


FIG. 2-3 Energy of activation for the diffusion of atoms: x_1, x_3, equilibrium positions (lattice sites); x_2, middle position

When the temperature of the specimen is raised to T_2, the mean energy of the majority of atoms, E_2, goes up to E_3, and that of the minority to E_4. Locally, the temperature within the lattice may be so high that the crystal may even vaporize. Then an atom may free itself from the action of the forces around it — now it will be free to move about. If an atom moving at a high velocity comes near an open surface, it may escape into the surroundings (sublimation). As is seen, for an atom to be able to move through the lattice, it must be activated. The activation energy for the diffusion of atoms depends on the magnitude of the energy barrier, Q (Fig. 2-3) which is in turn determined by the strength of bond between atoms. Diffusion proceeds gradually; the elementary displacement, x_i, of an atom in the lattice is a few angstroms. From state to state, atoms move in jumps.

Diffusion might proceed by four mechanisms.

1. Exchange of places between two adjacent atoms (Fig. 2-4*a*). This would involve the expenditure of a large amount of energy because each atom must move a distance of two atomic diameters, and the ensuing local distortion of the lattice would be appreciable. Therefore this mechanism should be ruled out.

2. Motion of interstitial atoms (Fig. 2-4*b*). The atom at lattice site *1* moves to an interstice *2*. Its further motion will be through the interstices *3*, *4*, *5*, and *6*. For this to happen, an appreciable amount of energy must be imparted to the atom. Therefore, this mechanism can exist in the case of heterodiffusion in solid solutions where an interstitial atom is far smaller than an atom of the parent metal.

3. Circular exchange of four atoms (Fig. 2-4*d*). Now four atoms at a time move around a ring. This mechanism is most of all likely to exist in metals with a closely packed lattice.

4. Motion of vacancies (Fig. 2-4*c*). Now vacancies in the lattice change places with adjacent atoms. The motion of vacancies is in a direction opposite to that of atoms. On taking up the vacancy at *1*, atom *2* clears room for atom *3*, and atom *4* can now move into the site previously occupied by atom *3*, leaving behind a vacancy.

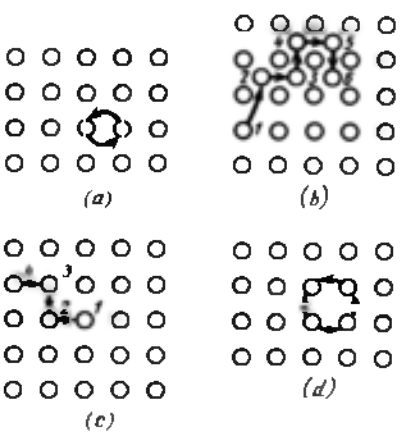


FIG. 2-4 Mechanisms by which atoms move in a metallic lattice

As has been found theoretically, the activation energy for self-diffusion in copper should be 1 008 kJ mol^{-1} for the exchange mechanism, 966 kJ mol^{-1} for the interstitial mechanism, 269 kJ mol^{-1} for the vacancy mechanism, and 378 kJ mol^{-1} for the ring mechanism. The experimental figure is 210 kJ mol^{-1}. Hence, of the four likely diffusion mechanisms, pure metals and alloys with a substitutional solid-solution structure show preference for the vacancy mechanism. With it, an atom at a lattice site may jump to an adjacent site. For this to happen, the diffusing atom should be activated thermally so that it can clear the potential barrier between the sites, equal to Q_j, the activation energy for vacancy jump. The probability of a jump is a function of the equilibrium concentration of vacancies, C_v, in the metal such that

$$C_v = C \exp(-Q_f/RT) \tag{2-2}$$

where Q_f is the activation energy for vacancy formation, C is a constant, $R = 8.315$ kJ mol^{-1} is the gas constant, and T is the temperature in kelvins.

The activation energy for diffusion is usually taken as the sum of the activation energy for vacancy formation, Q_f, and the activation energy for vacancy jump, Q_j.

So far we have only considered vacancies. Solids are, however, abundant in other lattice imperfections, called dislocations. The rate of diffusion through the dislocation core is a function of Burgers' vector and may be very high. Grain boundaries, too, may be treated as lattice imperfections. The observed activation energy for diffusion along grain boundaries is about half the figure for diffusion by the bulk mechanism. The surface migration of metallic atoms shows a low activation energy as well. By Fick's first law, the rate of diffusion is defined as the quantity of material, q, diffusing across an interface plane of unit area, dA, in unit time, dt, through a distance, dx. The quantity of diffusing material, q, is a function of the concentration gradient of the element involved, dc/dx, in a direction normal to the interface (Fig. 2-5) and is proportional to the diffusion coefficient, D, or mathematically

$$q = -D\, dc/dx \tag{2-3}$$

The "−" sign on the right-hand side of the equation indicates that the diffusion proceeds from a region where the concentration of the element is high towards a region where its concentration is low. Numerically, the diffusion coefficient is the weight of material diffusing across unit area in unit time under unit concentration gradient ($dc/dx = 1$).

Variation in concentration with time is defined by Fick's second law

$$\partial c/\partial t = D(\partial^2 c/\partial x^2) \tag{2-4}$$

FIG. 2-5 Flow of atoms across an element of section

It is assumed that the diffusion coefficient is independent of concentration. As the temperature is raised, the rate of diffusion increases exponentially

$$D = D_0 \exp(-Q/RT) \tag{2-5}$$

where D_0 is the frequency factor depending on the type of lattice and the oscillation frequency of the diffusing atom, Q is the activation energy for (or heat of) loosening in kJ mol^{-1}, $R = 8.315$ kJ mol^{-1} is the gas constant, and T is the temperature in kelvins. The quantity Q characterizes the binding energy of atoms in the lattice. The larger its value, the greater the energy required to loosen the lattice or, which is the same, the higher the energy required for an atom to jump from a site to another site or an interstice. The activation energy is a maximum in the case of bulk diffusion, Q_b, and is smaller than the activation energy for boundary diffusion, Q_{bound}, and the activation energy for surface diffusion, Q_s, that is

$$Q_b > Q_{bound} > Q_s$$

Diffusion proceeds predominantly over the surface or along the boundaries of grains and mosaic blocks because the lattice distortion and lattice imperfections (vacancies, dislocations, stresses, and cracks) are most pronounced there. The rate of diffusion along grain boundaries depends on the angle between the grain axis and the direction of diffusion. From the view-point of diffusion bonding, it is attractive to speed up diffusion by producing an excess concentration of vacancies through plastic deformation. In specimens of silver subjected to twisting, the rate of diffusion has been found to increase at least hundredfold. This increase is proportional to the rate of deformation, that is, the instantaneous concentration of vacancies. Excess vacancy concentration can also be produced by rapid cooling (quenching) or irradiation with highly energetic particles.

2.3 The Processes and Factors Responsible for Joint Formation in Diffusion Bonding

Several physico-mechanical processes usually take place in diffusion bonding at the same time. These are diffusion, recrystallization, creep, formation and motion of dislocations, formation and motion of vacancies and interstitials. Each can, to a varying degree of accuracy, be evaluated by the following well-known equations

$$A_1 = B_1 \exp(-Q_1/E_0) \tag{2-6}$$

$$A_2 = B_2 + C_2 \exp(-Q_2/E_0) \tag{2-7}$$

.....

$$A_i = B_i + C_i \exp(-Q_i/E_0)$$

Here, Q_i is the activation energy for the respective process, and E_0 is the thermal energy imparted to the test specimen. Their ratio appears as an exponent in all equations and is a dimensionless quantity which may serve as a similarity criterion for the processes involved.

Thus, with regard to diffusion bonding, it is not enough to limit consideration only to the thermal quantities associated with a particular physico-mechanical pro-

cess. Account must always be taken of two factors, mechanical energy and thermal energy, because the two always come into the picture at the same time.

Let M be the factor related to the mechanical energy expended to deform a volume V. Then in terms of the ratio of bonding pressure, p, to the yield strength, σ_y, it can be defined as

$$pV/\sigma_y V = M \tag{2-8}$$

Similarly, let N be the factor related to the thermal energy imparted to the same volume. Then, in terms of the ratio of the instantaneous temperature, T, to the melting point, T_m, it can be defined as

$$T\gamma cV/T_m\gamma\, cV = N \tag{2-9}$$

where γ is the density of the metal, and c is its specific heat.

Suppose that the two factors may be added together. Then Eqs. (2-7) and (2-8) can be re-arranged to yield the equation of a line in straight segments

$$\frac{p}{(M + N)\,\sigma_y} + \frac{T}{(M + N)T_m} = 1 \tag{2-10}$$

The above form of relation between the bonding pressure, p, and the bonding temperature, T (the dashed line in Fig. 2-6) does not actually occur in diffusion bonding. The actual relation is $p = f(T)$; it is the same for all pressure welding processes (the solid line). The relation holds when joints made at various temperatures and pressures and also with different methods of heating and different holding times (durations of pressure) and temperatures have about the same strength. The relation

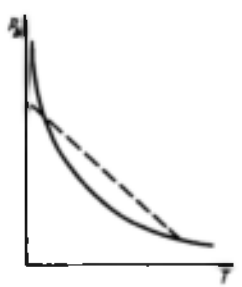


FIG. 2-6 A plot of pressing load versus bonding temperature in pressure welding

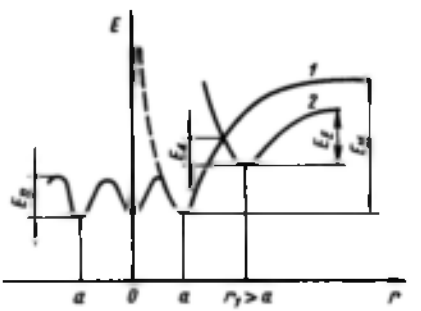


FIG. 2-7 Variations in the potential energy of interaction between metallic crystals in diffusion bonding

E_E, energy of electrostatic (van der Waals) interaction; E_M, energy of dissociation of a metallic bond; E_A, energy of activation for the formation of a metallic bond; E_D, energy of activation for diffusion; a, constant of a metallic lattice; r, distance of approach

is a hyperbolic curve, especially at low temperatures and high pressures. If we consider the mechanical and thermal energy factors together, then

$$MN = pT/\sigma_y T_m \tag{2-11}$$

Hence the hyperbolic relation between p and T has the form

$$pT = MN\sigma_y T_m = \text{constant} \tag{2-12}$$

An examination of the various pressure welding processes on the basis of Eq. (2-12) will show that pressing load and bonding temperature are interrelated.

In practice, the surface of solids always shows asperities and is covered with a hard-to-remove layers of adsorbed gases, water and other substances which must be removed so as to make a sound and strong joint. As already noted, the joint will gain in reliability and strength, if the weld zone is expanded into the bulk of the material owing to the self-diffusion and the interdiffusion of atoms in the specimens being joined.

For ease of analysis, it is convenient to break up the diffusion bonding operation into phases or steps. There are two basic phases terminating in an energetically steady state for the atoms at the surfaces of the specimens, and also in completion of all physical processes in the bond zone. As is shown in Fig. 2-7, when crystals with clean surfaces are brought together to within a distance comparable with the lattice constant, it is more advantageous energetically for metallic bonds to form (curve *1*). Because of the presence of adsorbed layers, however, the potential energy of atoms on the real surfaces of polycrystals may prove insufficient for metallic bonds to form. Therefore the initial contact interaction will be associated with the nonsteady (fluctuating) electromagnetic fields of the metal specimens and of the adsorbed layers on their surfaces (curve *2*). The activation by heat and deformation

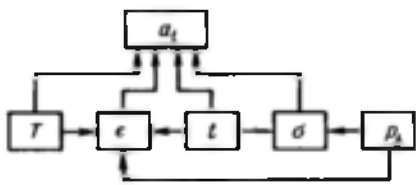
TABLE 2-1 Analysis of Diffusion Bonding

Phase	Effects leading to formation of joint	Form of diffusion	Activation parameters
1. Formation and development of an actual contact between the surfaces	Deformation of asperities, diffusive levelling of asperities and machining ridges	Surface diffusion (and bulk diffusion at $T = 0.5\ T_m$), self-diffusion	T, p
2. Formation of a joint and development of bulk interaction	Formation of metallic bonds between groups of atoms, sealing of voids, reduction in defect concentration, stress relaxation in the bond zone	Bulk and grain-boundary self- and heterodiffusion, motion of dislocations, physicochemical state of the surfaces	T, ϵ (p, t, σ) composition of medium

and the scouring of the surfaces raise the potential energy by E_A, so that metallic bonds (a joint) can form. In actual diffusion bonding, the initial contact is first established within isolated areas; it is then followed by the spread of the contact into the bulk of the material. A phase-by-phase analysis of diffusion bonding is given in Table 2-1.

As is seen, the second phase (and also the first phase in the case of high-temperature creep, $T > 0.5\ T_m$) is characterized by bulk, grain-boundary and surface hetero- and self-diffusion. It is therefore legitimate to think that the bond is formed by the diffusion mechanism. The principal parameters activating diffusion bonding in vacuum are bonding temperature, bonding pressure and deformation (ϵ), with the process proceeding in time t.

Specimens in low-carbon, low-alloy, and stainless steels diffusion bonded in various combinations at $T = 1023\text{-}1423$ K, $p_b = 10\text{-}100$ MPa, $\epsilon = 1\text{-}20\%$, and $t = 0.5\text{-}60$ min in a vacuum of not over 2.6×10^{-3} Pa have been tested for impact strength, a_i, and the results analyzed on a computer. From the constraint equations thus derived, the parameters involved have been found to be related as shown below:



Since $\epsilon = f(\epsilon, t)$ and $\epsilon = \psi(p_b, t, \sigma)$, regression analysis yields sufficiently simple expressions for the weldability criterion, K_w. On their basis and considering the high-temperature ultimate tensile strength, σ_t, we can readily determine the thermal and deformation parameters of diffusion bonding, optimal in terms of the bond quality. As an example, for a joint with $a_i = 0.5$,

$$K_w = (\sigma_t^1/p_n)(0.284 + p_n/25 - 0.046\sqrt{t}) \qquad (2\text{-}13)$$

where σ_t^1 is the tensile strength of the softer metal in the combination at the bonding temperature T, p_n is the nominal bonding pressure, t is the holding time (duration of pressure), K_w is the minimum value of σ_t^1/σ_t^2 (where σ_t^2 is the ultimate tensile strength of the harder steel) at which the joint will have the desired impact strength, a_i. The impact strength of the joint increases in proportion to $\sqrt{t}$, which is characteristic of diffusion-controlled processes.

A simple, single model that would cover all the varieties of diffusion bonding is difficult to develop. At best, it can include one or several combinations out of the multitude of metallurgical and mechanical effects. The concrete conditions for diffusion bonding and its results will vary according to which of the effects predominate.

When the bonding pressure or the bonding temperature is sufficiently high, the asperities on the mating surfaces undergo a marked plastic deformation until the surfaces conform to each other's contour almost ideally. The resultant joint is fairly strong, because atomic bonds have formed within some areas. When the bonding pressure is low, it takes a longer time to make a joint comparable in strength. The pressing step breaks up any thin films, and plastic deformation proceeds at the juvenile surfaces.

In cases involving materials differing in mechanical, physical and chemical properties, a quality diffusion bond can best be made with the use of an interlayer of some other material. This technique, when used judiciously, substantially expands the field of application for diffusion bonding in vacuum. Depending on the materials to be joined, the interlayer may serve more than one purpose. When the materials differ in the thermal coefficient of linear expansion (TCLE), residual stresses are likely to come into being in the bond zone as the pieces cool off, these stresses being the larger, the greater the difference in TCLE. In the circumstances, it is advantageous to use an interlayer having an intermediate value of TCLE and possessing high plasticity — it will cause the existing stresses to relax. Good results could be obtained with interlayers whose TCLE varies gradually with the height of the composite. At present, a technique has been developed by which a composite can be diffusion-bonded in vacuum with several interlayers.

In joining multicomponent materials mutually immiscible in solid state it pays to use interlayers promoting bulk diffusion in the contact area. Above all, they should be capable of forming solid solution with both materials being joined. In contrast, barrier interlayers should be used between materials likely to form intermetallic and other brittle phases so as to minimize or avoid altogether bulk diffusion. Materials for barrier interlayers should be such that the diffusion flow into the parent materials is higher than in the reverse direction. Then they will retain their barrier properties for a long time even when heated to a high temperature. Interlayers can also be used in joining materials which do not form brittle phases in the bond zone. Now their purpose is to promote the actual contact between the mating surfaces, the formation of metallic bonds, and to speed up diffusion processes. Interlayers have proved effective in cases where the joint must be free from residual macroplastic deformation.

The processes occurring in the material-interlayer-material system are complex in nature. In each particular case they are determined by the properties of all the constituents in the composition, their purity, the method of application and thickness of the interlayer, welding conditions, etc. The thickness of an interlayer in relation to that of the materials being joined has a direct bearing on the contact effects (hardening, softening) and the form of the stressed-strained state arising in the weld zone when an external load is applied. For ease of fabrication, it is therefore essential to clean the mating surfaces, to prevent their re-oxidation, to apply a sufficient bonding pressure and heat, and to hold the joint for an ample span of time.

Vacuum diffusion bonding is almost unlimited in scope. In practice, however, one should maintain optimal conditions for a diffusion bond to form. This necessitates a deep insight into the effects and factors examined above.

2.4 Mating Surfaces

When treated or finished by some physico-mechanical means, the surface of a solid can be described in terms of its geometry and its physical state.

The term "geometry" refers to the profile mainly produced by machining (such as turning, milling, grinding, etc.). It may be classed into macrogeometry (waviness) and microgeometry (roughness). In the case of an ideally clean solid, the profile can be resolved into a macroprofile and a microprofile, each depicted by a curve, a waviness curve and a roughness frequency curve.

Roughness can widely vary in shape, height, and spacing between asperities. It is customary to model asperities as pyramids, cones, and spherical projections. The degree of roughness also varies according to the form of machining applied. For example, roughing with a grinding wheel leaves asperities 40 to 120 μm high, turning and planing 20 to 40 μm, and polishing 0.3 to 1 μm.

Waviness is a form of macrogeometrical deviations (which also include out-of-planeness, convexity, taper, etc.). It refers to a periodic sequence of high and low spots close in dimensions and spaced closer apart than other forms of macrogeometrical irregularities. Ideally, a waviness profile (or profile tracing) is a sinusoid.

As already noted, roughness varies according to the type of machining applied, the kind of tool used, and the material of the specimen. As an example, Fig. 2-8 shows profile tracings, I, for an aluminium and a steel surface after turning. Tracings II through IV illustrate how the asperities are gradually deformed by gauge blocks at various pressures. On comparing the thickness of transparent oxide films (not over 3×10^{-2} μm thick) with the height of the asperities, it is readily seen that the width of the tracing is hundreds of times larger than the thickness of the oxide film on a freshly scoured (machined) surface.

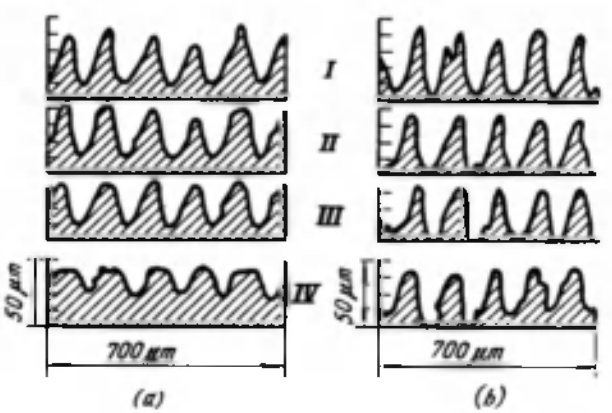


FIG. 2-8 Profile tracings for (*a*) aluminium and (*b*) steel pieces after turning

In diffusion bonding, the geometry of the surfaces to be joined determines the area of the actual contact between them. Obviously, the contact area increases as the surface finish is improved.

The physical state of the surfaces to be joined depends on the presence and composition of surface films and, especially, the structure of the surface layers of the material. An ideally clean (juvenile) surface, free from oxide film and adsorbed layers of liquid and gas molecules, can only be obtained in a high vacuum (around 1.3×10^{-6} Pa). Then a cloud of free or roaming electrons exists above the metal surface. These electrons alternately escape from and return to the metal. As a result, the surface is covered by a double electric (or dipole) layer in which the "—" side is the electron cloud, and the "+" side is the holes in the surface layer (left by escaping electrons). The density of the surface charge varies from area to area and depends on its geometry. The potential is at its highest at the peaks of asperities.

An ideally clean surface can exist for a very short interval of time (a small fraction of a second) at a fracture or immediately after machining. In air, all low and high spots acquire a film of oxides instantaneously, to which are added adsorbed layers of water, gas and grease molecules. As an example, let us consider the formation of an adsorbed monolayer under a variety of conditions. From the kinetic theory of gases it is known that the number, n, of molecules hitting unit area (1 cm^2) of the container per unit time (1 s) is given by

$$n = 3.52 \times 10^{22} p_a / \sqrt{MT} \qquad (2\text{-}14)$$

where p_a is the air pressure, Pa, M is the relative molecular mass, and T is the temperature in kelvins. Hence it is an easy matter to show that at atmospheric pressure every square centimetre of the container's surface will be hit by 5×10^{22} oxygen molecules per second. This is about 10^8 times as great as is necessary to form a monolayer on the surface. The time necessary for a monolayer to form at a surface temperature of 293 K and several pressures is given in Table 2-2.

As follows from Table 2-2, exposure of a metal surface to the surrounding air at atmospheric pressure instantaneously results in the formation of a monolayer.

TABLE 2-2

Pressure, Pa	Time, s	Pressure, Pa	Time, s
101 080	2.4×10^{-9}	1.3×10^{-5}	18
13 300	1.8×10^{-8}	1.3×10^{-6}	180
1 330	1.8×10^{-7}	1.3×10^{-7}	1.8×10^{3}
1.3	1.8×10^{-4}	1.3×10^{-8}	1.8×10^{4}
1.3×10^{-3}	0.18	1.3×10^{-10}	1.8×10^{6}
1.3×10^{-4}	1.8	1.3×10^{-12}	1.8×10^{8}

A real surface always displays a complex system of adsorbed layers. As a rule, the juvenile surface is covered by oxides strongly bonded to the metal. Their thickness may be several tens of angstroms. Depending on the actual conditions, the oxide film is overlaid by adsorbed layers of gases and water. The outer surface may also be covered by adsorbed layers of polar and nonpolar molecules of organic substances (grease, oil). Grease layers may be fairly thick.

The thickness and sequence of surface layers depend on the composition of the surrounding medium. The layer next to the pure metal is ordinarily an oxide film (Fig. 2-9). This is a loose cover just beginning to adapt itself to the crystal structure of the metal. As the oxide film gains in thickness, its structure approaches more and more that of a regular lattice, and its rate of growth slows down because the oxide film itself serves now as a barrier for the motion of metallic cations and oxygen molecules towards one another. The oxide layer remains negative relative to the metal at the interface. The outer layer of the oxide film turns electrically positive and adsorbs the electrically negative layer of oxygen which can no longer form a bond because there are no free metallic cations present. As a result, the oxidized metal (see Fig. 2-9) acquires two double electric (dipole) layers. This is how the electric bonds are typically arranged at the surface.

Apart from oxides, the metal surface is always covered by grease and gas molecules and water vapour which form coatings widely differing in thickness. For example, water vapour forms a film 50 to 100 molecules thick, and grease produces still thicker coats. After degreasing with petrol, the remaining layer of organic substances may be 1 to 5 μm thick. Even a very careful treatment with solvents leaves a grease film 10 to 100 molecules thick. It is practically impossible to remove all of the grease from the metal surface with any solvent, because the adsorptive

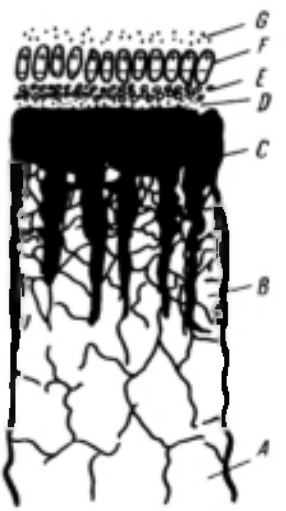


FIG. 2-9 Surface of a metal in air

A, metal layer not plastically deformed; *B*, surface layer of completely unoriented crystals with oxide interlayers; *C*, oxide layer (the "+" and "−" signs denote the characteristic polarity of internal and external boundaries and of the upper layer); *D*, adsorbed layer of oxygen ions and neutral air molecules; *E*, water molecule layer; *F*, grease molecule layer; *G*, ionized dust particles

bond between the grease molecules and the metal atoms is augmented by an electric one. Polar grease molecules form a double electric layer on the metal, and this is responsible for the very strong bond between the metal and the monolayer. Grease molecules show one more important property — they readily penetrate any microcracks on the metal surface. On finding their way into microscopic crevices, these grease monolayers tend to split up the cracks still wider. At the tip of cracks about 100 μm wide this splitting pressure of grease molecules may approach the yield strength of the metal. Characteristically, metal specimens thoroughly cleaned but then soiled by handling cannot be joined by diffusion bonding, because the grease layer thus left on the surface is more than one molecular layer thick. When the surface has been mechanically cleaned (by grinding with a stone or emery cloth, by turning or milling, etc.), it will some time afterwards come by an oxide film even in a dry air. The thickness of the film is given in Table 2-3.

TABLE 2-3

Metal	Film thickness, μm	Time, s
Copper	3×10^{-3}	30
Aluminium	12×10^{-4}	15
Iron	2×10^{-3}	40
Molybdenum	2×10^{-3}-3×10^{-3}	40
Germanium	2×10^{-3}-3×10^{-3}	80

Subsequently, the oxide film grows very slowly, if at all. Its growth, however, can be stimulated by heating the specimen or by activating, in one way or another, the surroundings. The thickness of oxide films is difficult to estimate by eye. It has been found that invisible, that is, sufficiently transparent oxide films on machined surfaces are no more than 0.03 μm thick. Temper colours on steel parts constitute a coat 0.04 or 0.5 μm thick, and visible scale is over 0.5 μm thick.

The physical state of the thin surface layers can be substantially modified by machining and will usually vary according to the degree of deformation (strain) entailed. The depth of strain hardening, for example, is determined by the degree of strain and the nature of the material. As an example, Table 2-4 lists data on the surface hardening of steel parts subjected to several forms of machining. In the table, the degree of strain hardening is the ratio of the microhardness at the surface and in the bulk of the specimen.

Grinding leaves the surface layer saturated with structural defects (dislocations, vacancies, and the like), so it is in a specific physical and chemical state. This state has of necessity an effect on both the properties of the layer and the kinematics of the processes that can take place on heating. As is seen, insight into the physical state and structure of the surface layer calls for knowledge of many interrelated factors.

TABLE 2-4

Form of working	Degree of strain hardening, %	Depth of strain hardening, μm
Turning on a lathe	120-150	30-50
Milling	120-160	40-100
Drilling and counterboring	160-170	180-200
Surface grinding	150	16-35
Lapping with pastes	112-117	3-7

2.5 Cleaning and Shielding of Metals in High Vacuum

The complex structure of the surface layer can be disturbed by any one of several physical factors. For example, heating to a relatively low temperature can drive off the adsorbed water. Heating to a high temperature in a shielding atmosphere or in a glow discharge can vaporize oxide films. In diffusion bonding, however, it is essential not only to remove any contaminants present, but also to prevent recontamination of the surfaces being joined. In this respect, vacuum holds out a special promise.

The properties of vacuum as a shielding medium depend on the amount of residual gases present in the welding chamber. It is instructive to compare the amount of impurities present per unit volume of commercial shielding media and vacuum. This is done in Table 2-5 which gives the oxygen and nitrogen content of the medium as a function of rarefaction (vacuum). On comparing the data, it may be concluded that a low vacuum (around 133 Pa) is a better shielding medium than commercially pure argon carrying 0.05% O_2 and 0.23% N_2. The diffusion-bonded joints made in a vacuum of 13.3 Pa are better than those made in especially pure argon carrying 0.003% O_2 and 0.03% N_2.

When the workpiece is heated in vacuum, its oxides, nitrides and hydrides are caused to dissociate and vaporize. Hydrogen is especially easy to drive off.

TABLE 2-5

Pressure, Pa	Gas content per unit volume, %		
	Total	O_2	N_2
101 080	100	20.1	79.9
133	0.13	0.03	0.1
13.3	0.013	0 003	0.01
1.3	0.0013	0.0003	0 001
1.3×10^{-1}	0.00013	0.00003	0.0001
1.3×10^{-2}	0 000013	0.000003	0.00001

Characteristically, gases and their compounds are removed not only from the surface. Given certain conditions (time and temperature), their percentage is also reduced in the bulk of the material. This substantially improves the physico-mechanical properties of the metal.

When a low-alloy or a carbon steel is heated to over 973-1073 K, observable oxidation is non-existent at a vacuum not over 1.3×10^{-2} Pa. Given the same vacuum and temperature, a steel alloyed with 1-2% Cr acquires a thin oxide film visible in a microscope. To avoid this, the vacuum must be brought down to 1.3×10^{-3} Pa. A still better vacuum will be required for a higher chromium content.

The vacuum in the welding chamber required for a particular metal or alloy can approximately be determined from the following considerations. The solubility of diatomic gases (oxygen, nitrogen, and hydrogen) in metals at pressures below those required for a second phase to form is given by a well-known equation

$$S = Rp^{1/2} \tag{2-15}$$

where S is the solubility of the gas in wt %, R is a constant, and p is the gas pressure in pascals.

If we know the limiting solubility of a gas in a metal at atmospheric pressure and the required final gas concentration in the metal, we can estimate the partial pressure of the gas in the welding chamber necessary to give that concentration:

$$S_1/S_2 = (p_1/p_2)^{1/2} \tag{2-16}$$

where S_1 is the solubility of the gas at atmospheric pressure, S_2 is the final gas content of the metal, p_1 is the residual pressure of the gas, in Pa, and p_2 is the residual gas pressure at which the metal will not be oxidized (in Pa).

As an example, let us take up commercial titanium. The limiting solubility of oxygen in titanium at atmospheric pressure (p_1 = 10 180 Pa) is 0.18 weight fraction (18%). The allowable oxygen content of titanium is assumed to be not over 0.0015 weight fraction (0.15%). On substituting these values in Eq. (2-16), we get the critical partial pressure of oxygen, p_2 = 6.65 Pa. In terms of air pressure, this is 33.25 Pa.

At present, the degree of vacuum at which no oxide film will form on the surfaces being joined can only be determined by experiment.

As it breaks up and dissociates upon heating in vacuum, the oxide film can partly be removed by the diffusion of some oxides into the bulk of the material (one metal dissolves in another), or it can be converted to species having a substantially smaller surface area.

2.6 Mechanical Contact Between Clean Mating Surfaces

The pressure applied to the clean mating metal surfaces causes them to establish a contact. At asperities the pressure exceeds their yield strength, and the surface crystals are brought together to within the atomic distance in the lattice. This is how the actual contact is established.

A distinction of the state in which metal crystals reside at the contact is that they

can instantaneously seize and form a continuous crystalline structure within the area of the actual contact.

The concept of *actual contact* is a broad one. The point is that, when brought together to within a distance of the order of atomic spacing, the surface crystals will not always produce a permanent and continuous crystalline structure. Therefore, it should be assumed that the actual contact may be separable or monolithic.

Actual contact contrasts with *mechanical contact* which refers to a separable junction where the surface layers can be from fractions of a micrometre to fractions of a millimetre thick. An *electric contact* is any mechanical contact which can conduct an electric current. A *bond* (or *joint*) *contact* is any mechanical, separable junction converted by pressure and diffusion into an actual, permanent contact. For mechanical contacts (Fig. 2-10), the actual surface area is given by

$$A_a = n\Delta A_a \tag{2-17}$$

where A_a is the total actual area of the contact, n is the number of elementary asperities, and ΔA_a is the area of an elementary (unit) microcontact determined by the size and shape of elementary asperities. The other symbols used in Fig. 2-10 are as follows. A_c is the contour area of the contact within which all the elementary asperities are found. This is a macroscopic quantity determined by the applied pressure, p, and the waviness and roughness of the mating surfaces (Fig. 2-11*a* through *d*). A_n is the nominal area determined by the size of the mating parts. (For example, if the mating parts are bars, this will be their cross-sectional area.) In some cases, the mating surfaces are ground to each other so well that the contact is formed by both the smooth and the rough areas. Then, $A_c = A_n$ (Fig. 2-11*a*).

According to experimental data [6], an increase in the bonding pressure applied to the rough mating surfaces in the cold state causes a faster increase in the number of microcontacts, but not in their mean area.

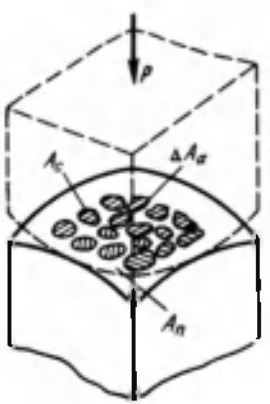


FIG. 2-10 Mechanical contact between metal surfaces

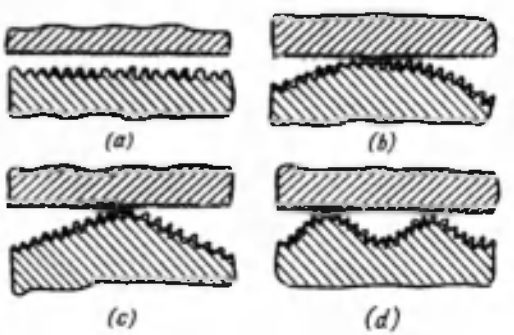


FIG. 2-11 Contact between wavy and rough metal surfaces

In the case of a bond contact, we should consider not only the bonding pressure, but also the average temperature in the plane of contact. Where elastic and elasto-plastic contact is involved, a high bonding pressure gives rise to plastic deformation rising with time. This rise will continue until the applied load is balanced by the resistance at the points of contact. In other words, this rise will continue until the product of the actual contact area, A_a, by the term related to the yield strength of the material in compression, σ_y, becomes equal to the applied load, P. On re-arranging we get

$$A_a = P/C\sigma_y$$

where C is the rigidity factor of the asperities.

At room temperature (293 K), the ratio of the actual contact area to the nominal area decreases with increasing load.

Load, N	0.3	2.0	10.0	30.0
A_a/A_n	1/170 000	1/10 000	1/700	1/130

2.7 Plastic Deformation and Pressing Load in Diffusion Bonding

As is currently believed, in the pressure welding of metals and alloys the pressing load is responsible for several effects. Firstly, it breaks up and removes the brittle surface oxide film and contaminants. Secondly, it brings the mating surfaces close together, thereby enhancing contact and atomic interaction. Thirdly, it activates the subsequent bulk processes (diffusion, recrystallization).

In the case of diffusion bonding in a vacuum or an inert gas, where the purity of the atmosphere is very high and the pieces are not practically oxidized on heating, there is no need for a deformation that would give rise to a marked flow of metal. Now, deformation is only needed to crush the asperities on the mating surfaces so that a successful actual contact can be formed. More specifically, the purpose of the pressing load is mainly to produce a microplastic deformation which would lead to a maximum contact area between the mating surfaces and promote interdiffusion. On the mating surfaces, the shape of the interdiffusion front is determined by their surface geometry: as the quality of surface finish deteriorates, local zones of high diffusion rate come into being and persist on moving away from the plane of contact

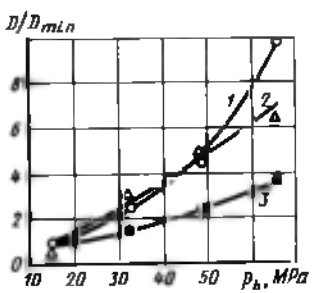


FIG. 2-12 Diffusion coefficient in the near-contact zone as a function of pressing load. The distance from the contact interface is as follows: (1) 5 μm; (2) 10 μm; (3) 15 μm

(Fig. 2-12). In the narrow zone next to the plane of contact, the value of the diffusion coefficient may vary with the quality of surface finish, but the difference is minimal. On moving away from the plane of contact the diffusion coefficient varies with the depth of the near-contact zone differently, depending on the surface treatment used: after electropolishing, the rate of diffusion falls off faster; after grinding, the associated acceleration of diffusion can be observed at a considerable depth.

2.8 Thermal Properties of Materials. Heating and Diffusion Treatment at Constant Temperature

The atoms in crystals are in a constant vibratory motion about their equilibrium positions in the lattice. The amplitude of the motion is about 3% of the atomic distance. As the temperature of the material is raised, the amplitude of the motion goes up while its frequency remains unchanged. At the melting point of the material, the amplitude of the vibratory motion is 12% of atomic spacing. The increase in the amplitude raises both the kinetic and the potential energy of the atoms, and this brings disorder into the atomic structure in the case of metals. In a crystal of N atoms, each atom has three degrees of freedom for translational motion. A crystal as an entity has three degrees of freedom for translational motion and three for rotational motion. So, the lattice has $(3N - 6)$ degrees of freedom for vibratory motion.

As already noted, the amplitude of atomic vibration in a crystal increases with rising temperature. By quantum theory, the energy corresponding to frequency ν is given by

$$E = \frac{1}{2} h\nu + \frac{h\nu}{\exp(h\nu/RT) - 1} \tag{2-18}$$

where h is Planck's constant in kJ s, R is Boltzmann's constant in kJ K^{-1} mol^{-1}, and T is the absolute temperature in kelvins.

The first term in Eq. (2-18) is the zero energy (the energy existing at zero degrees absolute). The second term is the temperature-dependent part of the energy. At low temperatures, the energy E and the associated heat capacity, dQ/dt, become negligible. Since the second term gives the mean energy, E_1, of a linear oscillator, the energy of a gram-atom having $N = 3$ degrees of freedom is $E = 3NE_1$.

Now let us introduce the characteristic temperature, $\Theta = h\nu/R$. When $T > 0$, the energy of one mole of a crystal containing N atoms is

$$E = 3NR \frac{\Theta}{\exp(\Theta/T) - 1} \tag{2-19}$$

and at $N = 1$,

$$E = 3R \frac{\Theta}{\exp(\Theta/T) - 1} \tag{2-19a}$$

According to Ya. I. Frenkel, the spacing, r, between the species with rising temperature changes as shown in Fig. 2-13. For each temperature or the correspon-

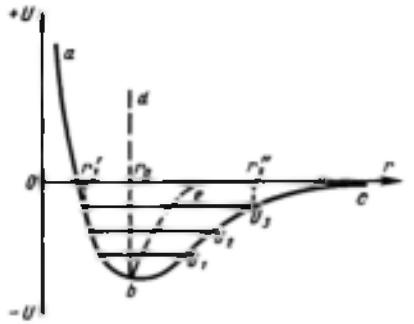


FIG. 2-13 Thermal expansion of metals

ding energy level, $U_1 - U_3$, the mean distance between particles varies along the curve *be*. As is seen, at temperature T_3 corresponding to energy U_3, the spacing r_3' is the shortest distance between the atoms, and r_3'' is the longest distance. The curve *abc* (Fig. 2-13) is asymmetrical about the line *bd*. The curve *ab* is steeper than the curve *bc*. Hence we may conclude that the species in a solid are in a non-harmonic vibration. This means that the departure of a particle from the equilibrium position, r_0, to the left is different from that to the right. The swing to the right is larger than to the left, because the forces of attraction build up more slowly than the forces of repulsion. This is why an increase in temperature is accompanied by an increase in the mean spacing between the centres of adjacent atoms — a fact which explains thermal expansion.

The thermal coefficient of linear expansion is found from the slope of the curve *bc* relative to the vertical. As is seen, the slope of the curve is continuously increasing from a point corresponding to one temperature to another point corresponding to a higher temperature. Therefore, we may conclude that the thermal coefficient of linear expansion increases with rising temperature. The mean TCLE is given by

$$\alpha = \frac{l_2 - l_1}{T_2 - T_1} \frac{1}{l_1} \tag{2-20}$$

where l_1 and l_2 is the length of the specimen at T_1 and T_2 respectively. The true TCLE

$$\alpha_t = \frac{dl}{dT} \frac{1}{l_t} \tag{2-21}$$

The thermal coefficient of volumetric expansion is $\beta = 3\alpha$. This equality does not hold for polycrystals and single crystals of any crystallographic habit, except cubic. Metals having a cubic lattice expand isotropically on heating; metals having a noncubic lattice frequently expand anistropically on heating.

The bonding energy of atoms in a crystal may be evaluated with sufficient accuracy indirectly, using the heat of vaporization, the thermal coefficient of linear expansion, the compressibility factor, and the activation energy for self-diffusion. The higher the bonding energy, the higher the melting point (although there are a few exceptions), and the lower the thermal coefficient of linear expansion and the compressibility factor.

The most important outcome of heating a solid is the increase in the rate of diffusion. This goes for both self-diffusion and heterodiffusion. The rates of physical and chemical conversions which proceed by a diffusion mechanism can be increased many times through a relatively small rise in temperature. A sharp increase in temperature speeds up the re-distribution of atoms as individual areas on the mating surfaces are brought close together, promotes removal of various lattice defects in the bond zone, and raises the rate of diffusion. It has been found experimentally that at low temperatures and a low rate of diffusion the resultant bond is anything but perfect. On the other hand, an excessive rise in temperature leads to high rates of diffusion and grain growth when similar metals are being joined, or the formation of an intermetallic layer when dissimilar metals are being joined. Both factors impair the quality of the diffusion-bonded joint.

On heating to a temperature which lies below the point of recrystallization but is sufficient to bring about the diffusion of atoms and molecules, there will be difficulties associated with removal of oxides and contaminants from the mating surfaces, the deformation of asperities will be hampered and, what is most important, there will be a slow progress in recrystallization responsible for the formation of shared grains in the bond zone of similar metals — a factor essential for a sound joint to be obtained.

According to A. A. Bochvar, the recrystallization temperature, T_r, of pure metals is related to their melting point in such a way that $T_r = 0.35T_m$ to $0.45T_m$. Alloying raises the recrystallization temperature to $0.6T_m$-$0.8T_m$ of the alloy. The metals nickel, cobalt, and iron melt at 1 728 K to 1 808 K, and their recrystallization temperature lies anywhere between 693 K and 723 K. For nickel-base high-temperature alloys with a melting point 1 623 K to 1 673 K the recrystallization temperature lies anywhere between 973 K and 1 273 K. This is an indication that alloying can substantially slow down diffusion processes, including those associated with recrystallization. By analogy with Bochvar's relation, the optimal temperature of diffusion bonding, T_b, is a certain fraction of the melting point of each of the metals being joined, so that T_b/T_m = constant. As experimental data show, this ratio is anywhere between 0.53 and 0.86. The optimal temperature for diffusion bonding is believed to be $0.7\ T_m$. If unwanted structural transformations are likely to occur, the bonding temperature should be brought down and the holding time increased in proportion. From recent studies it can be concluded that the bonding temperature may be brought down to as low as $0.2T_m$-$0.4T_m$ for a number of metals, no matter which crystal structure they have. In such a case, however, it is essential to clean the mating surfaces thoroughly, to raise the vacuum to anywhere between 1.3×10^{-5} and 1.3×10^{-6} Pa, to increase the pressing load, and to extend the holding time (duration of diffusion treatment at constant temperature).

2.9 Analysis of Diffusion-Bonded Joints

The choice of techniques for the analysis of diffusion-bonded joints is governed by the nature of the effects involved and the state of the art. Most frequently, these techniques are metallography, electron microscopy, spectral analysis, X-ray

microanalysis, X-ray diffractometry, tracer analysis, microhardness measurement, determination of mechanical properties at low and high temperatures, tests for long-term strength and creep, determination of thermal stability and corrosion resistance, etc. Whatever the technique used, it should be able to resolve localized features. For proper evaluation of the diffusion-bonded zone, several techniques may be used at a time.

Metallographic analysis brings out the number, size, shape, relative position, and relative amounts of phases and constituents. Unfortunately, the small size of the transition zone coupled with a marked difference in mechanical properties, and the difference in electrochemical properties — all this hampers the investigation of the zone with an ordinary light microscope. The problem is somewhat alleviated when the specimens are prepared at low rates of polishing with hard abrasive wheels, and by thermal etching. Yet, the smallest size of a feature that can be resolved is limited by the resolving power of the microscope whose magnification is never larger than 1 500 diameters.

An ordinary electron microscope has a far better resolution (a magnification of up to 40 000 diameters). Yet, replicas and foils can be prepared with difficulty, if at all, especially in cases involving the transition zone between some dissimilar metals. A better performance is shown by a scanning (or flying-spot) electron microscope. In it, a sharply focused electron beam, or probe, scans the surface of interest to produce an electronic raster, or scan. Electrons, either reflected or secondary (those emitted due to the excitation of the material by primary electrons),generate a signal. Variations in the signal on the surface of the specimen form an electron shadow of that surface. The scanning reflection electron microscope has overcome the limitation of light and transmission electron microscopy because it has a depth of focus which is 300 times that of a light microscope. A further advantage is that no replica or foil has to be prepared. Practically any metallographic specimen or a fracture up to 12 mm in diameter and up to 3 mm in thickness can be investigated in a scanning electron microscope at a magnification of 20 000-30 000 diameters. Combined with metallographic analysis, the instrument gives an all-round picture of what is happening during diffusion bonding. The high magnification is effective in bringing out the traces of plastic deformation which takes place at substantial pressing loads, transitions from one orientation to another in the bond zone, and the structural detail of the diffusion layer and parent metals.

Changes in the structure upon heating, cooling and holding can be studied with high-temperature metallographic units. Those made in the United States (such as the Unitron), Japan (the Union), and Austria (the Reixert) include a vacuum chamber and a heater. The chamber is set up on an ordinary metallographic microscope fitted with a special-purpose long-focused lens. Soviet-made units in this class come complete with a microscope and a device to stretch the specimens. The practical magnification is up to 500 diameters. The equipment permits bright-field, dark-field, oblique-light and phase-contrast microscopy.

Microhardness measurement is likewise widely used in studies of diffusion processes. With it, the investigator is in a position to estimate the depth of the diffusion zones, the resultant phases and constituents having a characteristic microhardness

(such as intermetallides, eutectics, and the like). The smallest size of the indentation that can be conveniently viewed is 5 μm. With a smaller indentation, the error may be as large as 20% of the indication. In special cases, one can, after the indentation diagonal has been measured, prepare replicas for viewing in an electron microscope. This can improve the resolution down to 1 μm. However, the technique cannot be used to analyze joints between dissimilar metals because of the strain hardening of the specimen's surface, which cannot be removed. A remedy in such a case is to use a load and an indention diameter which would minimize strain hardening.

An improvement upon the above technique is the use of microthermo-emfs induced in a circuit made up of two conductors with their two junctions maintained at different temperatures. The ratio of the measured thermo-emf to the temperature difference is indicative of the nature of the conductors. One of the conductors is a tungsten needle, and the other is an area on the specimen. The area may measure from 5 to 30 μm across, depending on the hardness of the specimen. This technique is convenient in obtaining tentative data.

Absorption X-ray micrography depends for its operation on the difference in the absorption of monochromatic X-rays by various elements as the radiation is made to pass through a thin specimen. The absorption coefficient is independent of the state in which a given element is present (in solid solution or as a chemical compound). The absorption of X-rays is proportional to the concentration of the element. At certain wavelengths, characteristic of each element, the absorption coefficient changes stepwise. If a specimen in which the concentration of the element of interest varies from area to area is placed on a photographic plate and illuminated with a beam of monochromatic X-rays at certain wavelength, the difference in X-ray absorption will be revealed on the plate upon development. X-ray micrography can be of the contact type and the projection type. The latter has a resolution of 0.5 μm. The plate to be X-rayed is 80 to 140 μm thick. It is effective in the analysis of microinhomogeneities in metals, the qualitative and quantitative distribution of elements in the transition zone, and any inhomogeneities in the bond zone.

X-ray microanalysis utilizes the fact that a beam of highly energetic electrons is focused by a system of electron lenses to a micron diameter (an electron probe) and directed onto an area of the specimen viewed in a light microscope at a magnification of 400 to 600 diameters. Striking the specimen within the area probed, electrons excite an X-ray emission which is analyzed by a system of crystals into a spectrum, and this is registered by counters. The wavelength and intensity of X-ray emission give clues for qualitative and quantitative analysis. The sensitivity of the technique varies from element to element, and its accuracy depends on the excitation conditions and the chemical composition of the specimen because the intensity is a nonlinear function of concentration. For precise measurements, resort is made to standards; with them the accuracy is 5% or better, whereas without standards it is 10% to 15%. The instruments currently in use are capable of analyzing the elements from magnesium to uranium in areas measuring down to 2-5 μm across. Some instruments are capable of determining boron, nitrogen, and other elements. Almost all microanalyzers are fitted with a scanner with which one can both analyze elements at a point and obtain a topographic picture of element distribution at

several characteristic wavelengths and also in reflected and absorbed electrons. The technique offers wide scope for the analysis of the structure and composition of the transition zone. A fundamental advantage of the technique is that it is capable of investigating the transition zone in multicomponent systems with the best attainable resolution.

The phase composition of the transition zone in diffusion-bonded joints can be investigated with an X-ray diffractometer. This is done by comparing diffractograms of the materials being joined before and after bonding. The lattice constants can be precisely determined with feedback X-ray units.

A convenient way to determine the composition of the transition zone is by means of layer-by-layer spectral analysis, using the technique developed at the Fundamental Vacuum Diffusion Bonding Research Laboratory (PNIILDSV) on the basis of the procedure proposed by the Spectral Analysis Laboratory of the Paton Electric Welding Institute (Kiev, USSR). This form of analysis is applicable to cases where the width of the transition zone is over 40 μm.

Tracer analysis (radioautography) offers a means for investigating diffusion processes at the interface of the materials being joined and establishing the pattern of changes in the diffusion flow at the plane of the initial contact and in the bulk of the bond zone. In this analysis, a suitable radioactive element is introduced into the materials to be joined, and its diffusion is then monitored.

Mechanical testing yields reliable data on the strength and ductility of diffusion-bonded joints at temperatures not over 293 K where time is not a factor. At higher temperature, time must be taken into consideration because materials grow less stable and undergo plastic deformation which changes the shape of the specimens. The values of strength and plasticity obtained by hot tests strongly depend on the duration of a test, the loading rate, and the temperature at the time of testing. Because of this, the values obtained from short-duration tests at 293 K cannot be used to design parts intended to operate under a sustained load and at elevated temperatures. Now whether the material and its joint are fit for use is ascertained by special performance tests. The performance of joints at elevated temperatures is determined by a set of tests related to their high-temperature strength and resistance. These include tests for creep, short- and long-term strength and ductility, fatigue strength at elevated temperatures, scale resistance, cyclic tests under alternating loads and fluctuating temperature.

Still other methods exist for the analysis of diffusion-bonded joints. Their choice, as already noted, depends on the composition and properties of the metals being joined and the specific features of the joints made. For example, joints between nonmetals and metals may involve petrographic analysis, infrared spectroscopic analysis, etc.

In the diffusion bonding of gray ferrite-pearlite cast iron it has been found that recrystallization affects not only the metallic matrix. The graphite plates in one of the two pieces appear to be a natural extension of those in the other piece. There is no chilled zone, the metal in and next to the weld is a monolithic cast iron, and the interface between the two is metallographically undiscernible.

The diffusion bonding of porous tungsten has shown that there is no interface between the pieces being joined — metallographic analysis has detected none. The

two tungsten pieces have formed a continuous single entity, and common grains have appeared in the bond zone.

No interface has been observed in diffusion-bonded specimens of a cermet (63% Mo and 37% Si). Microscopic analysis has revealed no physical interface between the pieces in diffusion-bonded joints involving T15K6 and BK8 cemented carbides[1].

To sum up, the diffusion bonding of similar materials under optimal conditions leaves no physical interface whatsoever between the pieces being joined, and the material undergoes no substantial changes in its physical and chemical properties.

The situation is somewhat different in the diffusion bonding of dissimilar materials. In the diffusion bonding of BK8 cemented carbide to 18X ГТ steel, there forms a transition layer different in properties and composition from the parent metals. The steel has a microhardness of 298, the cemented carbide a microhardness of 1 534, and the transition layer, 1 191. X-ray diffraction analysis shows that the lattice of the new phase is similar to those of W_3Co_3C and W_3Fe_3C. Thus, the diffusion bonding of dissimilar materials can produce transition layers or new phases consisting of the constituents of the parent materials.

A series of experiments has been conducted to elucidate the structure of the transition layers containing solid solutions. As has been found, on heating a specimen of Armco iron, which has a ferritic structure, is saturated with carbon from a steel-45 specimen. In turn, the area of the steel specimen adjacent to the joint is impoverished of its carbon. This can be seen from the increase in the amount of ferrite lying along the grain boundaries. The amount of ferrite in the steel-45 specimen gradually decreases on moving away from the bond zone. The manner in which the penetration of carbon varies with holding time (Fig. 2-14) is an indication that vacuum diffusion bonding can produce a full-strength joint only at a certain definite penetration. The holding time appears to affect both diffusion and self-diffusion.

In the diffusion bonding of commercial iron to a high-carbon steel (0.7% C), the carbon has been found to diffuse into a larger area over the same time interval. It is seen that a good deal of carbon has moved towards the carbonless specimen. Within the carbon diffusion zone chemical etching brings out the trace of the contact interface as a ferrite interlayer.

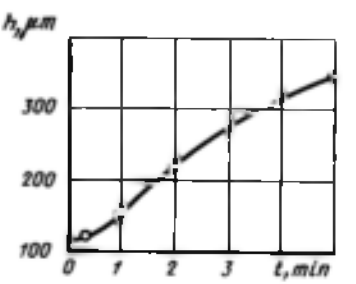


FIG. 2-14 Penetration (μm) of carbon into Armco iron as a function of holding time (duration of pressure)

[1] The designations of Soviet-made metals and alloys are explained in an appendix at the back of the book. — *Translator's note.*

Analysis of diffusion-bonded joints in high-speed steels (P18 and У7) to commercial Armco iron, both containing the same amount of carbon, has shown that the alloying elements in P18 steel have a marked effect on the diffusion of carbon.

One way to determine the extent of the diffusion zone is to measure the microhardness of a specimen cut out of the weldment across the bond. With this technique, a number of specimens have been examined, varying in carbon content. The experimental plots of microhardness in the bond zone as a function of holding time for, say, Armco iron and steel-45 (av. 0.45% C) agree well with theoretical curves showing the concentration distribution of alloying elements in the direction of diffusion in infinite space. The plots constructed from measurements are shown in Fig. 2-15. The data presented in this figure and also in Table 2-6 give an idea about the extent of the diffusion zone which varies with the chemical composition of the specimens, bonding temperature, holding time, and bonding pressure. The shaded areas between the joint line and the hardness plot in the bond zone are about the same in size. Hence it may be concluded that the technique is sufficiently reliable.

The plots in Fig. 2-16 likewise agree well with theoretical plots for the concentration distribution in the direction of diffusion in infinite space.

Metallographic analysis of joints between steel-20 (av. 0.20% C) and M1 copper, made by diffusion bonding in vacuum, shows that the surface of the joint is different in structure. Neither metallographic analysis nor X-ray microspectrometry can reveal any noticeable diffusion zone, and the bond occurs along grain boundaries.

From an analysis of joints in 12Х18Н10Т steel and Э10 silicon sheet steel it is seen that the former has a higher microhardness. On the Э10 steel side, grain growth is observed. This may be caused by high-temperature heating or recrystallization, because there is a slight permanent set. The width of the bond zone does not exceed the grain size. The joints have a high bending strength (σ_b = 2000-2600 MPa) and a high proof strength in twisting (σ_{tw} = 2250-4800 MPa).

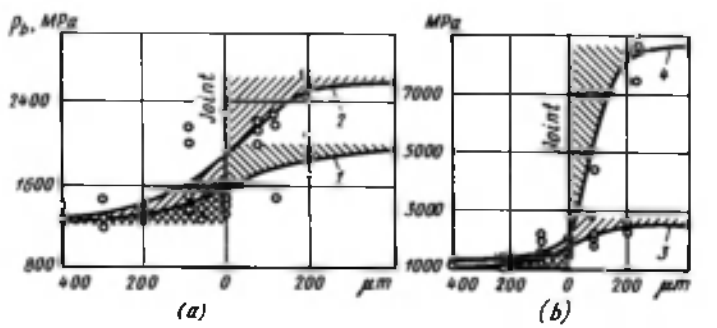


FIG. 2-15 Variations in microhardness in the diffusion bond zone

a: *1* — steel 45, *2* — У7 steel; *b*: *3* — Armco iron, *4* — P18 steel. The distance (μm) from the contact interface is laid off as abscissae

TABLE 2-6

Metals being joined	Bonding conditions			Microhardness (Vickers) at depth stated, μm				
				Armco iron		Steel-45		
	T, K	p_{cr}, MPa	t, s	300	100	0	100	300
Steel-45 + Armco iron	1273	20	30	137	192	192	257	257
			60	148	178	178	210	232
			120	146	163	176	176	232
			300	142	148	161	178	189
P18 steel + Armco iron	1273	20	300	139	168	213	511	925
V7 steel + Armco iron	1273	20		155	220	189	232	280
		1		123	158	199	321	321
	1073	10		139	174	210	255	286
		100	300	180	161	178	239	307
		1		125	129	140	260	296
	1173	10		125	142	163	296	296
		100		121	129	161	236	251

Where dissimilar metals are diffusion-bonded, interdiffusion gives rise to intermetallic phases. At the interfaces they form layers which interrupt the continuity of concentrations. The point is exemplified by a vacuum diffusion-bonded joint between specimens of Ст3 steel and aluminium, 20 mm in diameter and 30 mm high. On the microsection, the intermetallic layer stands out as a continuous white stripe separating the two parent metals. The original microhardness of aluminium is 25, that within 2.5 μm of the bond zone 72, and at the intermetallic interface, 2 120. The original microhardness of Ст3 steel is 107 Vickers, within 4μm it is 165 Vickers,

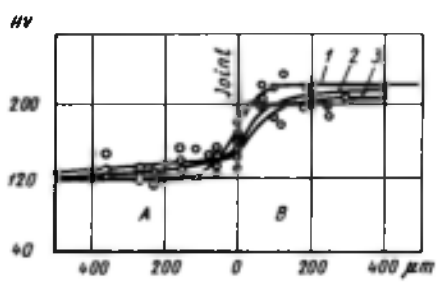


FIG. 2-16 Effect of holding time (duration of pressure) on microhardness in the diffusion bond zone between Armco iron (*A*) and steel 45 (*B*): (*1*) 0.5 min; (*2*) 1 min; (*3*) 5 min

and at the intermetallic interface, 336 Vickers. In an experiment, test specimens 6 mm in diameter showed a relatively low strength because of the presence of an intermetallic layer — they failed at a specific load of 40 MPa.

In another experiment, specimens of BT5-1 titanium alloy were joined to Armco iron at 973, 1 023, 1 073, 1 123, 1 173, and 1 273 K, and at varying bonding pressure and holding time. Metallographic analysis of the joints made at the minimum and maximum temperature showed that already at $T = 973$ K, $p_b = 17.6$ MPa and $t = 10$ min there was an increase in the hardness of the metal adjacent to the bond plane where a white intermetallic layer (0.5 to 0.9 μm wide) was formed. Its hardness could not be measured, nor could it be subjected to X-ray diffraction analysis. The microhardness of the iron adjacent to the interlayer raised to anywhere between 234 and 286, and that of the titanium alloy to anywhere between 428 and 490, the original microhardness being 300. It is believed that this increase in hardness is due to the interdiffusion of iron and titanium. At $T = 1\,273$ K, $p_b = 10.6$ MPa and $t = 100$ min, the intermetallic layer grew in width to 0.9-1.2 μm, but on the iron side there appeared a slightly etchable layer 3.0 to 3.5 μm wide with an elevated microhardness of 3 300 MPa. The microhardness of the titanium alloy adjacent to the interlayer also increased to 458-526. The presence of the brittle intermetallic layer led to the brittle failure of all test specimens. The specimens, even those with an ultimate tensile strength of 280 to 300 MPa failed at the bond plane with no plastic deformation at all. Hence it appears that the presence of brittle interlayers between the metals being joined can interfere with the formation of a full-strength joint.

The formation of an intermetallic layer can be prevented by using an interlayer of a third metal. For example, in the diffusion bonding of BT5-1 titanium alloy to Armco iron this may be molybdenum foil 0.3 mm thick. With this foil, specimens of BT5-1 titanium alloy and Armco iron, bonded at anywhere between 1 073 and 1 273 K, fail at the iron, with the molybdenum particles adhering to the titanium piece.

A feature characteristic of specimens bonded with a molybdenum interlayer is the presence, in the boundary iron zone, of a slightly etchable band 30 to 50 μm wide, which has an increased microhardness. Probably, the band is a solid solution of molybdenum in iron. As in the previous cases, the presence of a very hard interlayer between the molybdenum and iron results in a joint of poor strength. As an alternative, BT5-1 titanium alloy can be joined to Armco iron with a vanadium interlayer, because vanadium does not tend to form brittle compounds with titanium but does readily form solid solution with alpha-iron. The resultant bond is over the entire contact area, with the bond zone displaying a fairly high hardness which is an indication that no embrittlement has taken place. On the Armco iron side, there is a light-coloured band heavily alloyed with vanadium.

In the diffusion bonding of P18 high-speed steel to steel-45 (av. 0.45% C) with a copper interlayer 2μm thick at $T = 1\,073$ K, some of the copper diffuses into the steel-45 piece along ferrite and pearlite grain boundaries. Grains with a typically pearlitic structure can be seen at the bond on the steel-45 side. Because of the laminar structure of the pearlite, the bond zone shows a wavy appearance. Some of the copper diffuses into the steel-45 piece along ferrite plates.

It is especially difficult to diffusion-bond cemented carbides to ordinary steels because their thermal coefficients of linear expansion differ by a factor of 2. The resultant internal strains can be relieved through the use of interlayers. On heating, a cemented carbide piece is usually subjected to compressive stresses at the surface and to tensile stresses in the bulk. For this reason, one should never exceed the maximum permissible rates of heating. The rate of cooling must be one-eighth to one-tenth the rate of heating. As the thickness of the interlayer is increased, the internal stresses decrease. The decisive factor, however, is the material used. Good results have been obtained with interlayers from low-carbon steel, Permalloy, nickel foil, and powdered nickel. The latter has been found to produce a tight bond in weldments of У8 quality carbon steel and steel-45, on the one hand, and ВК-8 and Т15К6 cemented carbides, on the other. Along the bond line there can be seen oval tungsten carbide grains produced by diffusion and differing from the interlayer material, the cemented carbide and the steel pieces. As comparative tests have shown, the shear strength of a diffusion bond between a cemented carbide and a steel piece with no interlayer is 172 MPa, with an interlayer of powdered nickel, up to 260 MPa.

In the diffusion bonding of 18Х2Н4ВА steel to ВК20 tungsten carbide-cobalt alloy with a nickel foil interlayer (at $T = 1\,323$ K, $p_b = 10$ MPa, and $t = 10$ min), the boundary diffusion of some nickel into the steel produces a wedge-shaped expansion of the grain boundaries on the steel side. The diffusion zone in the solid solution is 20 μm wide.

A properly matched interlayer improves the strength and vacuum tightness of the bond, promotes the formation of compatible joints, produces a monolithic bond at lower bonding temperatures, reduces internal stresses within the bond zone, and prevents the formation of extremely stable oxides which interfere with diffusion on the surface of aluminium, titanium and precipitation-hardenable alloys. The interlayer diffuses into the parent material, thereby raising the melting point of the joint as a whole.

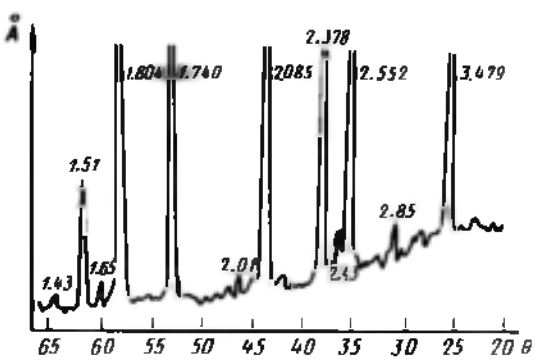


FIG. 2-17 Diffractogram of 22ХС cermet after interaction with МБ copper

Figure 2-17 shows a diffractogram of 22XC cermet diffusion-bonded to ME copper, recorded to detect a copper-bearing crystalline phase. As is seen, some of the interplanar distances are those associated with the lattice of copper spinel (copper aluminate), $CuO \cdot Al_2O_3$. There are no peaks associated with the lattice of pure copper.

X-ray diffractometry of joints in 5BMII niobium alloy diffusion-bonded to OT4 titanium alloy shows the presence of the brittle ω-phase and the residual β-phase on either side of the bond line. Also, there are blurred lines associated with the α-phase of titanium, which transpire through the β- and ω-phases. This usually occurs when a martensitic α-phase is formed on cooling. From the lattice constants of the observed phases and the results of X-ray diffraction microanalysis it can be concluded that diffusion processes play a detrimental role in that they promote the formation of brittle phases. It is, therefore, expedient to reduce the holding time and to increase the bonding pressure. In the bond zone, there is a predominant diffusion of titanium into niobium.

Bibliography

1. ARKHAROV, V. I. *Oxidation of Metals at Elevated Temperatures*. Metallurgizdat, Moscow, 1945 (in Russian).
2. AKHMATOV, A. S. *The Molecular Physics of Boundary Friction*. Fizmatguiz, Moscow, 1963 (In Russian).
3. BOKSHTEIN, B. S., BOKSHTEIN, S. Z., ZHUKHOVITSKY, A. A. *The Thermodynamics and Kinetics of Diffusion in Solids*. Metallurgiya, Moscow, 1974 (in Russian).
4. CHARUKHIN K. V. *et al. Bimetallic Joints*. Metallurgiya, Moscow, 1970 (in Russian).
5. GUEGUZIN, Ya. Ye. *Diffusion in Crystals*. Nauka, Moscow, 1974 (in Russian).
6. DEMKIN, I. B. *Contacting Between Rough Surfaces*. Nauka, Moscow, 1970 (in Russian).
7. KAZAKOV, N. F. *Diffusion Bonding in Vacuum*. Mashinostroyeniye, Moscow, 1968 (in Russian).
8. KAZAKOV, N. F. *The Formation of a Joint in Diffusion Bonding*. "Svarochnoye proizvodstvo", 1973, No. 9, pp. 48-50 (in Russian).
9. KAZAKOV, N. F. *Diffusion Bonding of Materials*. Mashinostroyeniye, Moscow, 1976 (in Russian).
10. KONYUSHKOV, G. V., KOPYLOV, Yu. N. *Diffusion Bonding in Electronics*. Ed. by N. F. Kazakov. Energiya, Moscow, 1974 (in Russian).
11. KOCHERGUIN, K. A. *Pressure Welding*. Mashinostroyeniye, Leningrad, 1972 (in Russian).
12. PATON, B. Ye. *The Welding of the Future*. In "Science and Mankind", vol. 3, Znaniye, Moscow, 1963, pp. 504-513 (in Russian).
13. SAMSONOV, G. V., BURYKINA, A. P., YEVTUSHENKO, O. V. *The Electron Mechanism of Diffusion Bonding*. "Avtomaticheskaya svarka", 1966, No. 10, pp. 30-34 (In Russian).
14. KHRENOV, K. K. *Welding, Cutting and Brazing of Metals*. Mashinostroyeniye, Moscow, 1973 (in Russian).

3

Principal Bonding Variables and Recommended. Procedures for Diffusion Bonding in Vacuum

N. F. Kazakov

3.1 Bonding Variables

Diffusion bonding is applicable to a large variety of metals, alloys, and nonmetals which can be joined in a diverse gamut of combinations or compositions.

From the experience accumulated to date, it is safe to say that most metals (such as nickel, copper, titanium and their alloys, and also steels) can readily be bonded together. About the same goes for the refractory metals molybdenum, tungsten, tantalum, and niobium. Molybdenum can easily be joined to steel and niobium. Quality diffusion-bonded joints can be made with ceramics, glass, quartz, semiconductors, graphites, cermets, cermets to metals. Cast iron can be joined to steel over a large surface. Dissimilar metals and alloys, such as titanium and copper, titanium and Kovar, titanium and Constantan, titanium and molybdenum, gold and bronze, silver and stainless steel, titanium and platinum, molybdenum and Kovar, aluminium and Kovar make good diffusion-bonded joints. What is important is that no other welding processes can make quality welds in the materials or the combinations listed above.

In diffusion bonding, the mating surfaces are heated to temperatures above or below the point of recrystallization under a small pressure. For this reason, metals and alloys can be joined without markedly affecting their physico-mechanical properties. One can readily control the progress of the bonding operation by varying the temperature of heating, the bonding pressure, the vacuum in the process chamber, and the holding time (duration of pressure), by using various forms of surface preparation, etc. If, however, one is to operate under optimal conditions, one should have a clear idea about the relationships between the above variables.

Graphically, the dependence of joint strength on bonding pressure can be depicted by a curve such as shown in Fig. 3-1. The bonding pressure is applied in order to secure a tight contact between the mating surfaces — a vital condition for the inter-diffusion of atoms of the metals joined. If the bonding pressure is below an optimal value, lack of bonding may occur at the centre line of the joint.

Variations in the vacuum in the process chamber and the bonding temperature in the course of diffusion bonding are shown in the plot of Fig. 3-2. It takes anywhere between 8 and 10 min to build up a high vacuum (portion *a-b*) in the chamber. Then comes the heating of the pieces, and the parts give up the gases dissolved in them. The quantity of outgassing depends on the analysis of the materials and the size of the workpieces. The rate of outgassing is further affected

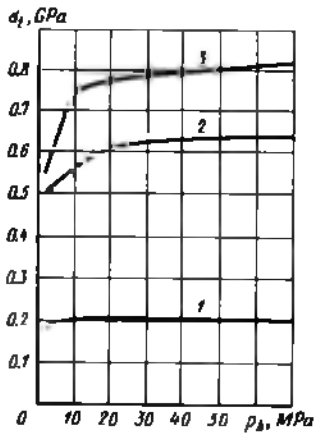


FIG. 3-1 Strength as a function of pressing load for joints made at $p_{vac} = 1.3 \times 10^{-2}$ Pa, $t = 5$ min
1 — СЧ 18-36 cast iron, $T = 1173$ K; *2* — steel 50, $T = 1373$ K; *3* — 45Х14Н14В2М steel, $T = 1473$ K

by the rate of heating. Outgassing raises the pressure in the chamber (portion *b-c*). The next step is degassing, or gas clean-up (portion *d-e*) during which the vacuum in the process chamber levels off to a steady-state value. Gas clean-up can be speeded up by raising the temperature of the pieces and by using a pumping system of a higher throughput. If this is not done, the vacuum in the chamber may rise to 0.13 Pa which is sufficient for ionization to take place, and for a break-down (a short-circuit between the coil turns) to occur. As a result of gas clean-up, the vacuum in the chamber is improved to 1.3×10^{-2} Pa (portion *e-f*). When this happens, the necessary bonding pressure is applied to clamp the pieces together. This is diffusion

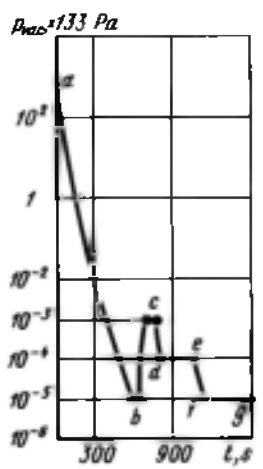


FIG. 3-2 Variations in vacuum in the diffusion bonding of T15K6 alloy to steel 45

bonding proper (portion *f-g*) which lasts 15 to 20 min. At the end of that time interval, heating is discontinued, the pressure of residual gases in the chamber is reduced, but the pressing load remains applied until the pieces have cooled to 303-423 K. If the load were removed earlier, the pieces might tear apart at the weld line in the case of dissimilar metals, or the strength of the weld might be impaired in the case of similar metals.

As follows from the plot in Fig. 3-3, for the diffusion bonding of T15K6 cemented carbide to steel-45 (curve *1*), copper to copper (curve *2*), Kovar to Kovar (curve *3*), the pieces should initially be heated to a temperature somewhat higher than the bonding temperature (by 323-343 K, portion *a-b*). This is because some of the heat is abstracted into the clamps. Also, some heat is lost as atoms of one metal diffuse into the other. At the end of the heating period the pieces should be cooled to a certain temperature in the chamber so as to avoid their oxidation in air and gas pick-up. For ferrous metals, the cooling temperature should not exceed 423-473 K (curve *3*), for nonferrous metals 353 K (curve *2*), and for cemented carbides 373 K (curve *1*). As the pieces cool to the temperatures indicated, each of the two materials contracts. With dissimilar materials, a steel piece will contract twice as much as a piece of cemented carbide or graphite. The resultant stresses in the joint can be reduced by bringing down the bonding temperature to a certain point (which is 1 173 K for, say, a joint in 12X18H10T steel and graphite). Below that temperature the relaxation comes practically to a stop. The stresses produced by the difference in contraction may cause the weld to crack at the bond plane or even to fail completely. This can be avoided by leaving the pieces under pressing load after the welding operation proper is completed. The load minimizes the mobility of dislocations which induces cracks in the field force. Also, when cooling proceeds under load the stresses are homogenized throughout the bulk, and cracking is thus prevented.

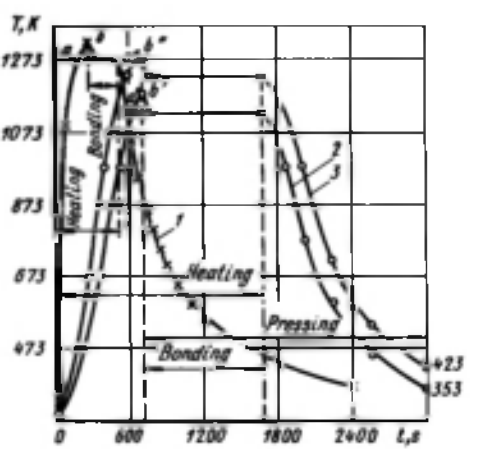


FIG. 3-3 Variations in temperature in the diffusion bonding of cemented carbide, copper and Kovar

SURFACE PREPARATION. The manner in which surface preparation for diffusion bonding can affect the joint strength can be evaluated experimentally. The usual procedure is to measure the strength of a diffusion bond at $T = 1\,273$ K, $p_b = 19.6$ MPa, and $t = 5$ min. In some specimens the mating surfaces are rough-turned; in others they are semi-finish turned; and in still others they are ground and polished. From an examination of the bar diagram in Fig. 3-4 it is seen that when the pieces are only rough-turned (bar *1*) the applied load is not enough to secure a maximum area of actual contact. The mating surfaces retain asperities which prevent an intimate contact at low pressing loads. In the case of semi-finish turning, the surfaces (bar *2*) are smoother, and the diffusion bond is usually 20% stronger. In the case of grinding (bar *3*), the bond strength is 10% lower than in the case of semi-finish turning. It is not unlikely that grinding leaves abrasive grains on the ground metal, and these interfere with diffusion processes. In the case of polished surfaces (bar *4*), the bond strength is about the same as in the case of semi-finish turning (bar *2*). This is an indication that the chosen variables (temperature, pressing load, holding time, vacuum) produce an intimate contact between the mating surfaces even with semi-finish turning.

It is very promising to clean the mating surfaces ultrasonically. When cleaning is done by ultrasound, the bending strength of diffusion-bonded joints rises from 686-784 MPa to 784-1274 MPa.

A straightforward procedure (Fig. 3-5) for evaluating the strength of diffusion bonds as a function of the manner in which the adsorbed material (oil, grease, dust, dirt, paint, etc.) is removed from the mating surfaces is by comparing them with undegreased surfaces (bar *1*) under identical conditions. Grease can be removed by rubbing with alcohol (bar *2*), acetone (bar *3*), and carbon tetrachloride (bar *7*). The oxide films can be best removed by pickling with acids (bar *4*) and alkalis (bar *5*). When pickled with an acid, the surface is smooth and covered by a uniform film

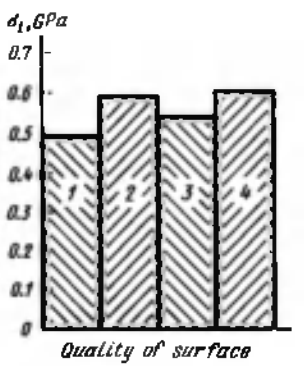


FIG. 3-4 Strength of joints in steel 45 as a function of surface preparation for bonding

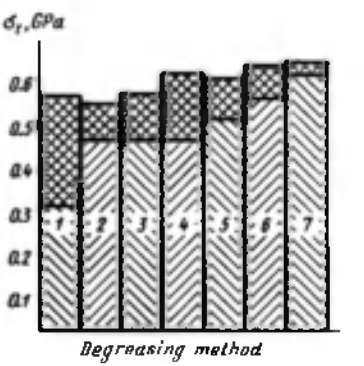


FIG. 3-5 Joint strength as a function of chemical treatment of the mating surfaces

over the entire surface area. Both adsorbed and oxide films can likewise be removed by heating in a vacuum (bar *6*). This is done at temperatures sufficient for the surface metal layers to vaporize and for the oxides to sublime. Thus treated, the surface layer has a higher activation energy.

From the foregoing, the following conclusions can be drawn.

(1) Surface preparation is of paramount importance for the actual contact to form.

(2) Semi-finish turning yields the best results.

(3) Chemical treatment of the mating surfaces stabilizes the strength of the bond, but different agents act differently. Degreasing with carbon tetrachloride (bar *7*) raises the strength by 14% as compared with degreasing with acetone (bar *2*) (Fig. 3-5).

(4) Vacuum cleans the metal surfaces and causes oxide films to sublime with the result that the joint strength is enhanced and the properties of the materials are improved.

BONDING TEMPERATURE AND BONDING PRESSURE (PRESSING LOAD). Of practical interest is the effect of these two variables in the temperature range 1073-1373 K and at bonding pressures of 4.9, 9.8, 19.6, and 49 MPa. In graphic form, the relationship is illustrated in Fig. 3-6, where it is assumed that the holding time is 5 min and the vacuum in the process chamber is 0.13 Pa. As is seen, the joint heavily depends on temperature at any bonding pressure. It follows from curve *1* (a bonding pressure of 4.9 MPa) that an increase of 100 K in temperature (from 1 073 to 1 173 K) doubles the joint strength, and the increase to 1 373 K trebles it. The effect is less pronounced at a bonding pressure of 9.8 MPa (curve *2*), when the rise in temperature from 1 073 to 1 373 K causes the joint strength only to double. At a bonding pressure of 19.6 MPa (curve *3*) the weld strength increases by a factor of 1.4, and at a bonding pressure of 49 MPa (curve *4*) by a factor of merely 1.2. At bonding pressures of 19.6 and 49 MPa there is an increase in the joint strength only

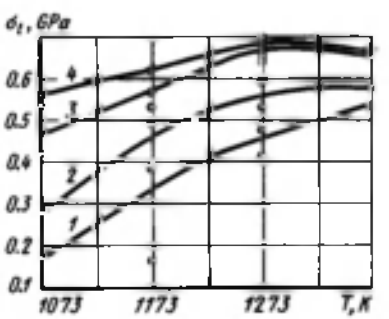


FIG. 3-6 Joint strength as a function of bonding temperature (at several values of pressing load)

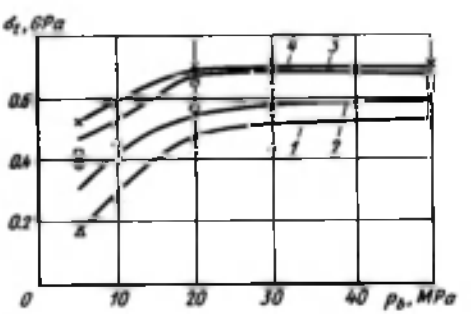


FIG. 3-7 Joint strength as a function of pressing load at several values of bonding temperature (t = 5 min)
(*1*) 1 073 K; (*2*) 1 173 K; (*3*) 1 273 K; (*4*) 1 373 K

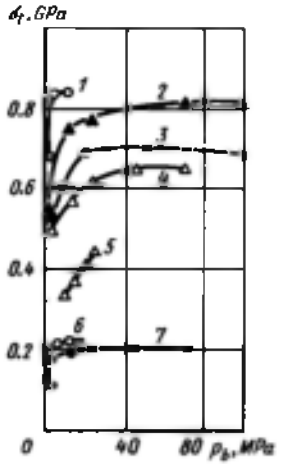


FIG. 3-8 Joint strength as a function of pressing load
1 — BT5-1 titanium alloy; *2* — 45X14H14B2M alloy; *3* — steel 50 (T = 1 273 K); *4* — steel 45 (T = 1 173 K); *5* — Kovar; *6* — M6 copper; *7* — CH 18-36 cast iron

at a temperature of 1 273 K. At 1 373 K it even goes down — a fact attributable to grain growth. The effect of grain growth is less noticeable at bonding pressures of 4.9 MPa and 9.8 MPa. Now the contact between the mating surfaces is not so intimate, and there is a marked spread in strength value at a lower strength level. The same goes for temperatures of 1 273 K and 1 373 K at bonding pressures of 4.9 MPa and 9.8 MPa.

As is seen from Fig. 3-7, the strength of diffusion-bonded joints goes up as the bonding pressure is raised from 4.9 MPa to 19.6 MPa at several values of temperature (1 073 K, curve *1*; 1 173 K, curve *2*; 1 273 K, curve *3*; and 1 373 K, curve *4*). The further increase in bonding pressure from 19.6 MPa to 49 MPa has only a slight effect on the bond strength. The explanation is that the mating surfaces are never perfectly smooth — they always have some degree of roughness. When such surfaces are brought together, they come in contact only at asperities accounting for a small share of the total surface area. As the pressing load is raised, the asperities are crushed, and the area of actual contact increases. Thus, the improvement in the bond strength as the bonding pressure is raised to 19.6 MPa and more is mainly attributable to the increase in the area of actual contact between the mating surfaces.

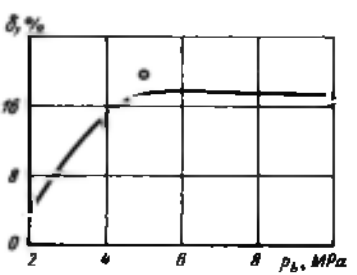


FIG. 3-9 Ductility (elongation) of joints in BT5-1 titanium alloy as a function of pressing load at T = 1 273 K and t = 5min

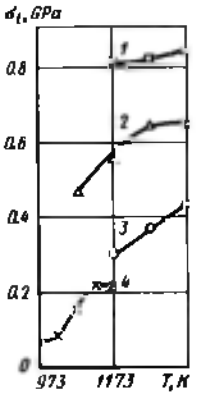


FIG. 3-10 Joint strength as a function of bonding temperature (*1*) BT5-1 titanium alloy; (*2*) steel 45; (*3*) Kovar; (*4*) M1 copper

The above point has been proved experimentally for a variety of materials. It has also been found that an increase in pressing load will raise bond strength up to a certain value. Any further increase in pressing load reduces it (Fig. 3-8). Experiments have demonstrated that the depth of the diffusion zone depends on pressing load in the same manner as the bond strength does (Fig. 3-9). This may be due to the fact that the pressing load equally affects diffusion and self-diffusion.

With all other conditions being equal, an increase in bonding temperature brings about an improvement in bond strength, but to a certain limit only. Any further rise in temperature will impair the strength owing to a rapid grain growth (Figs. 3-10 and 3-11). As follows from Fig. 3-12, bonding temperature affects both the impact strength (curve *a*) and percent elongation (curve *b*) of diffusion welds.

Thus, bonding temperature affects all the mechanical properties of welded specimens. This is illustrated in the diagram of Fig. 3-13 where the dependence is represented as a surface which intersects the p-T plane at the weldability boundary.

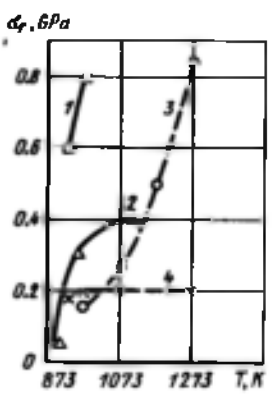


FIG. 3-11 Stress at fracture as a function of bonding temperature in stress-rupture tests
(*1*) OT4 titanium alloy + M1 copper (p_b = 4.9 MPa, t = 5 min); (*2*) OT4 titanium alloy + MB copper (p_b = 4.9 MPa, t = 10 min); (*3*) P18 steel + steel 45 (p_b = 19.6 MPa, t = 10 min); (*4*) MB copper + 22XC cermet (p_b = 9.8 MPa, t = 5 min)

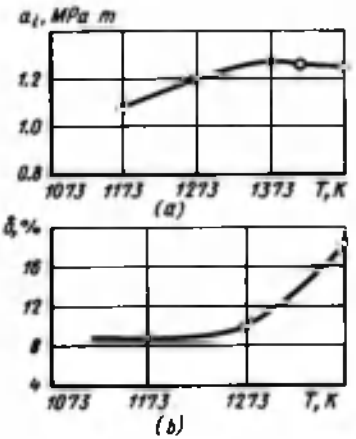


FIG 3-12 Plot of (*a*) impact toughness and (*b*) percent elongation of joints in BT5-1 titanium alloy as functions of bonding temperature

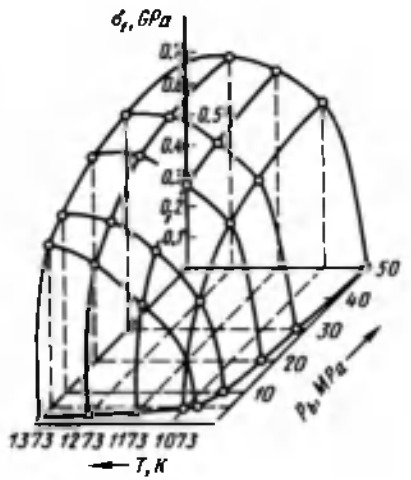


FIG 3-13 Strength of joints in steel 45 as a function of bonding temperature and pressing load

FIG 3-14 Specimens after tests (*a*) in tension; (*b*) twisting; (*c*) shear; (*d*) angle of bend; and (*e*) impa toughness

The points lying on the right of the boundary refer to conditions which will not produce a sound bond. The points lying on the left of the boundary refer to optimal bonding conditions. The horizontal plane relates the optimal bonding pressure to temperature. When the bonding temperature is equal to the melting point of the material, the bonding pressure is zero. When the welding operation is carried out at temperatures in the range 1073-1173 K and at bonding pressures of 4.9 MPa and 9.8 MPa, a full-strength joint is produced (it fails at the weld line), and the specimen has a low ductility. When a joint is made at temperatures of 1 273 K and 1 373 K and bonding pressures of 11.7 MPa and 49 MPa, it has the same strength as the parent metal, the interface between the two pieces is practically unnoticeable, and the specimens fail at the parent metal. At a bonding pressure of 49 MPa there is a fall in the ductility, which may be explained by strain hardening and grain growth. At a bonding pressure of 49 MPa the reduction in area is 4.5% and 16% at a temperature of 1 273 K and 1 373 K, respectively, whereas at a bonding pressure of 19.6 MPa the figures are 1.5% and 2%, respectively.

Similar relations between bond strength, bonding temperature, bonding pressure, vacuum, and some other factors exist for other materials.

Depending on the application of a weldment, its performance can be determined by tests for tensile strength, resistance to twisting, impact strength, bending angle, and shear strength (Fig. 3-14). The respective characteristics vary with bonding temperature and bonding pressure (Fig. 3-15).

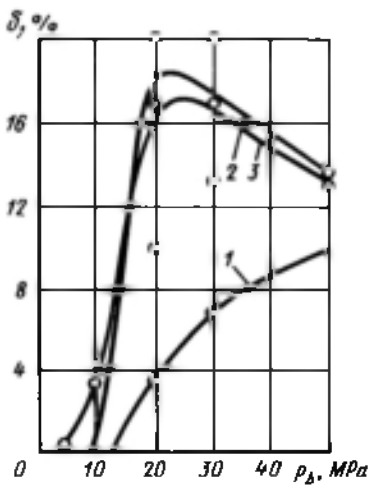


FIG. 3-15 Ductility (elongation) of joints as a function of pressing load and bonding temperature
1 — 1 173 K; *2* — 1 273 K; *3* — 1 373 K

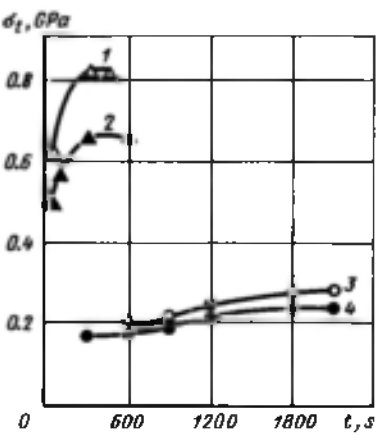


FIG. 3-16 Ultimate tensile strength (σ_t) of joints as a function of holding time (duration of pressure)
1 — BT5-1 titanium alloy; *2* — steel 45; *3* — Kovar; *4* — M1 copper

BONDING AND COOLING TIMES. As established by experiments, the dependence of bond strength on holding time is shown in the plots of Figs. 3-16 and 3-17. As is seen, the increase in holding time has a positive effect up to a certain limit. An excessive holding time, however, impairs bond strength owing to grain growth. About the same relationship exists between holding time, on the one hand, and percent elongation (see Fig. 3-17*b*), and impact strength (Fig. 3-17*c*), on the other. The data have been obtained by testing specimens vacuum-bonded at $T = 1\ 273$ K, $p_b = 19.6$ MPa, and $t = 5$ min. Figure 3-18 is a plot relating the strength of specimens cooled to various temperatures in the vacuum chamber. With the strength at 1 273 K taken as 100%, cooling in the chamber to 873 K improves the strength by 0.1%, cooling to 673 K by 33%, and cooling to 473 K by 44%. In the temperature range from 1 273 K down to 873 K ductility (elongation) is nil. The reduction in temperature down to 673 K raises the ductility to 3%, whereas cooling to 473 K brings the ductility up to 7%. When the temperature is brought down to 373 K and lower the ductility is a maximum, being 13%. To sum up, gradual cooling in the vacuum chamber under load enhances the strength and ductility of diffusion-bonded weldments.

VACUUM IN THE COURSE OF BONDING. The degree of vacuum is of primary importance to the normal progress of diffusion bonding. No less important are some processes accompanying the operation, notably the increase in outgassing from the workpieces and the chamber walls with increasing temperature.

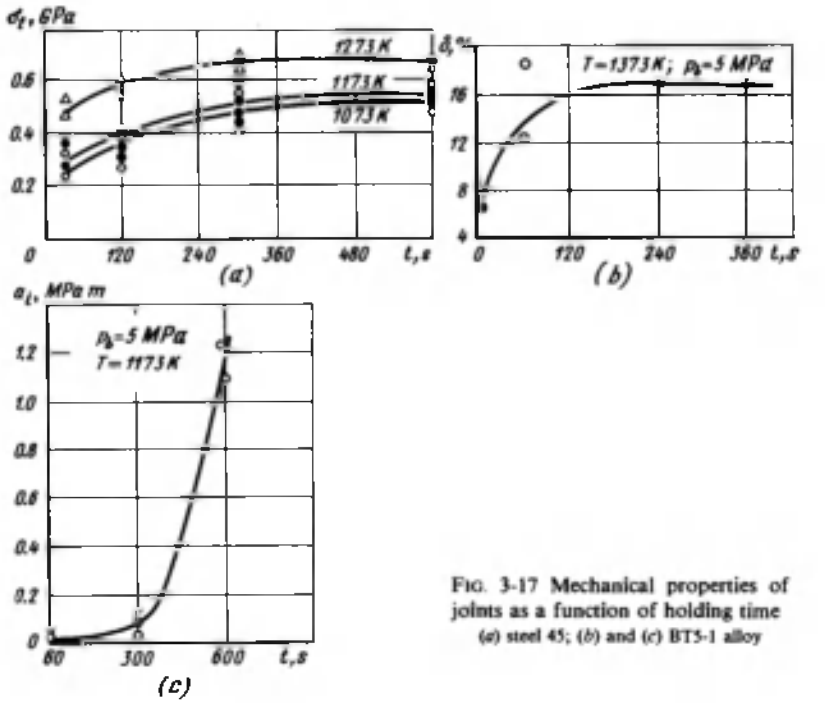


FIG. 3-17 Mechanical properties of joints as a function of holding time (*a*) steel 45; (*b*) and (*c*) BT5-1 alloy

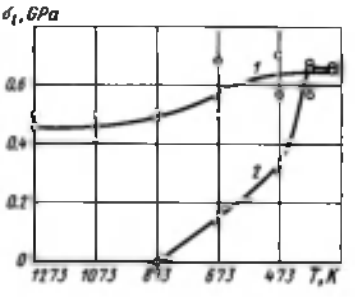


FIG. 3-18 Ultimate tensile strength, σ_t (curve *1*) and ductility, δ%, (curve *2*) of joints in structural steels as functions of cooling temperature

In experiments, this aspect has been investigated at bonding temperatures of 1 173 K and 1 273 K, a bonding pressure of 19.6 MPa, and a holding time of 5 min. As follows from the curves (Fig. 3.19) constructed for steel 45, at a temperature of 1 273 K (curve *2*) the bond strength improves markedly as the vacuum is built up to 13.3 Pa. As the vacuum is raised still more, there is only an insignificant improvement in bond strength. At a bonding temperature of 1 173 K (curve *1*) the bond strength goes up as the vacuum is raised to 1.3 Pa. With a further build-up of vacuum the gain in strength is less pronounced.

On a combined three-dimensional diagram the dependence of bond strength on temperature and vacuum can be depicted by a surface which intersects a horizontal plane to give the weldability curve (Fig. 3-20). Within the area at the right bottom, diffusion bonding cannot proceed.

When a hot metal is held in a vacuum, outgassing takes place. The first to be given up are the gases in the outer layers of the specimen. Then diffusion promotes the transfer of gases from the bulk of the metal towards its surfaces, and outgassing continues. The amount of outgassing from metals depends on the amount of gas dissolved, the temperature of the metal in the subsequent treatment, and the rate of diffusion which varies from metal to metal. Therefore, outgassing promotes the healing of microcracks at elevated temperatures, a factor substantially responsible for the high strength and ductility of diffusion-bonded welds.

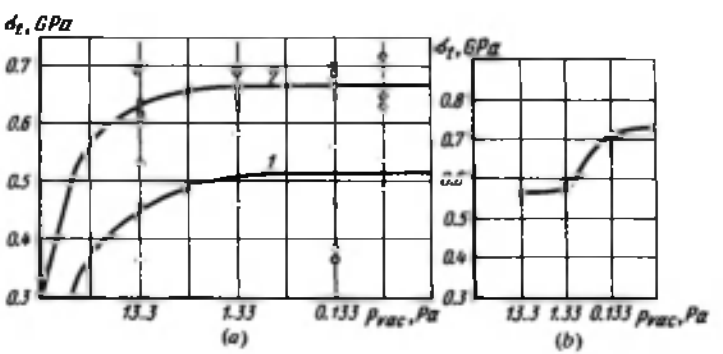


FIG. 3-19 Ultimate tensile strength, σ_t, of joints as a function of vacuum in the bonding chamber

(*a*) steel 45, p_b = 20 MPa, t = 5 min; (*b*) steel 45 + P18 steel, p_b = 4 MPa, t = 15 min, T = 1 223 K

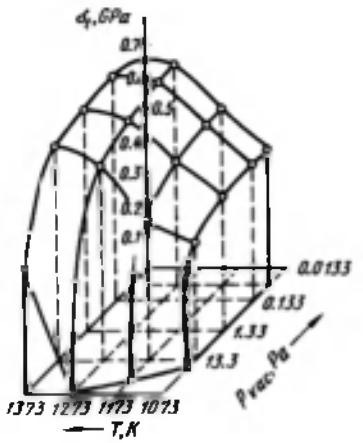


FIG 3-20 Strength of joints in structural steel as a function of vacuum and bonding temperature

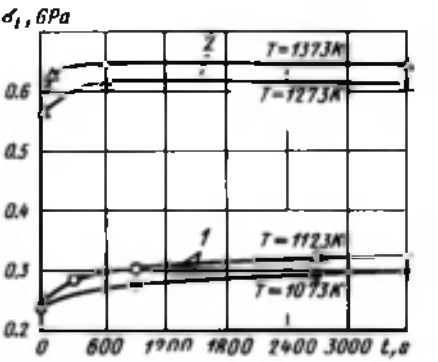


FIG. 3-21 Joint strength as a function of bonding temperature and heating time in vacuum

As calculations and experiments show, vacuum in the range 1.3-1.3 × 10^{-3} Pa is sufficient for the mating surfaces to be cleaned to the extent required to make a monolithic joint having the same strength as the parent metal. As is seen from curves *1* in Fig. 3-21, holding a copper specimen at elevated temperature in a vacuum for 15 min improves its strength by 20% to 25%. In the case of steel 45, vacuum treatment for 10 min improves the strength by 10% to 15% (curve *2*). It pays to extend the duration of vacuum treatment to a certain limit decided by the amount of dissolved gases and the thickness of surface films. With all other conditions being equal, the strength of a metal can be improved in the case of vacuum treatment by raising the temperature within a certain range. For example, the strength of diffusion bonds in 20X13 steel can be improved by 10% to 12% by heating them from 1 273 K to 1 373 K during 10 min, with the vacuum in the chamber maintained at 0.13 Pa, and cooling them to 473 K in the vacuum.

3.2 Procedures of Diffusion Bonding

In making a diffusion bond, it is essential to meet the following requirements.

(1) The mating surfaces must be thoroughly cleaned and a direct contact between them ensured.

(2) The materials should be heated to a temperature promoting diffusion across the mating surfaces over a specified time interval.

(3) The atmosphere around the joint should shield the material against oxidation or contamination or both.

To make a diffusion bond, the pieces to be joined are placed in a bonding chamber with their mating surfaces touching, a vacuum or any other controlled at-

mosphere is built up in the chamber, the pieces are heated to the specified temperature, a pressing load is applied, and the pieces are held compressed for a specified time interval. After the weld is complete, the weldment is held in the chamber or in the air for cooling, depending on the properties of the materials involved.

The optimal value of vacuum varies according to the properties of the materials and is chosen on the basis of previous studies and joint strength analysis. If the vacuum is not sufficient, the materials tend to be oxidized. If the vacuum is too high, the throughput of the equipment is reduced and the cost of the weldments goes up. A very high vacuum may only be warranted in cases where it is essential to clean the mating surfaces of oxides and various gases as effectively as practicable.

The mating surfaces should be machined so as to ensure a maximum area of actual contact. In choosing a method for surface preparation to the desired degree of roughness, the primary considerations are the technical and economic aspects of the operation.

Any surface films should be removed by a technique chosen according to the nature and form of films.

The bonding temperature should be chosen such that plastic deformation and diffusion processes can proceed at a high rate. Approximately, it ranges between $0.5T_m$ and $0.7\ T_m$ (where T_m is the melting point). For high-temperature and refractory metals the figure may be somewhat higher.

Bonding pressure should be sufficient for asperities on the mating surfaces to be crushed and for a maximum area of actual contact to be obtained. It can be calculated or determined by trial and error, depending on the type of materials to be joined. An optimal bonding pressure, that is, one which produces a sound weld and does not lead to macroscopic deformation in the bond zone, is approximately equal to the yield strength of the workpieces at the bonding temperature. Where dissimilar materials are to be joined, the choice of bonding pressure is decided by the weakest of the two materials.

Holding time is chosen according to whether an interlayer is used between the mating surfaces. This can be done by calculation or by trial and error.

The formation of a diffusion bond is accompanied by sublimation and dissolution of oxides, plastic flow, recrystallization, interdiffusion, etc., which may proceed concurrently or sequentially. Obviously, the observed speed of the bonding operation will be decided by the slowest process. An indirect indication that the processes occurring at the mating surfaces are complete is the ultimate strength of the bonded joint.

As an example, we shall follow the development of techniques and procedures for the diffusion bonding of ЭИ602 and ЭП99 high-temperature alloys.

BONDING WITHOUT AN INTERLAYER. Bonding pressure was raised from 9.8 MPa to 39.2 MPa in steps of 4.9 MPa. The welding operation was carried out at temperatures of 1 373 K, 1 423 K, 1 448 K, and 1 473 K. Holding time was set at 6 min. After bonding the specimens were withdrawn from the chamber and allowed to cool in the air. The bonding conditions were taken as optimal if the bonded joints had the same strength as the parent metal. For ЭИ602 alloy it was 735 MPa, and for

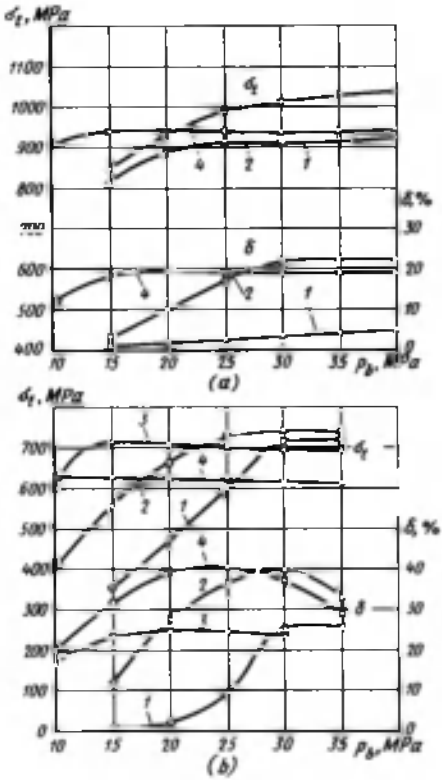


FIG. 3-22 Ultimate tensile strength and percent elongation of joints as functions of pressing load and bonding temperature
(*a*) ЭП 99 alloy; (*b*) ХН75МБТЮ (ЭИ602) alloy; *1* — T = 1 273 K; *2* — T = 1 423 K; *3* — T = 1 448 K; *4* — T = 1 473 K

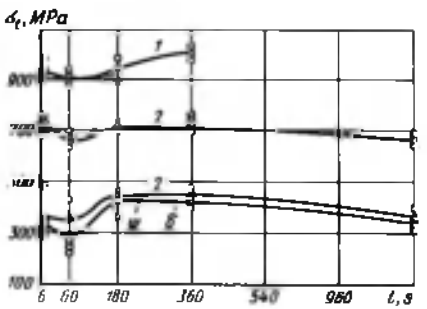


FIG. 3-23 Ultimate tensile strength, σ_t, of joints in (*1*) ЭП 99 alloy and (*2*) ЭИ 602 alloy as a function of holding time (duration of pressure). Bonding conditions: for ЭП99 alloy, T = 1 423 K, p_b = 39 MPa; for ЭИ 602 alloy, T = 1 423 K, p_b = 29.4 MPa

ЭП99 alloy it was 980 MPa. The arithmetic mean values of ultimate tensile strength, σ_t, and percent elongation, δ, as found from tests, are given in Figs. 3-22 and 3-23. At any temperature, an increase in bonding pressure led to an increase in the bond strength. Presumably this was primarily due to the increase in the area of actual contact, or the degree of completion of the first step in the operation. As metallographic examination showed, however, the first step appeared to reach completion at an instant different from that at which the bond matched the parent metal in strength. For example, when the bonding operation was carried out at a temperature of 1 373 K, actual contact was established over the entire area of the joint in ЭП99 alloy at a bonding pressure of 29 MPa; the strength was found to be 902 MPa. In the case of ЭИ602 alloy this occurred at a bonding pressure of 245 MPa and the bond strength was 588 MPa. Apart from an increase in the mean values of mechanical properties, the increase in bonding pressure resulted in a reduced scatter from the mean (a reduced variance).

In all probability, at the instant of actual contact the mating surfaces contained ultramicroscopic discontinuities and inclusions (not observed at a magnification of 450 diameters). Removal of the defects required some more time. As the bonding pressure was increased, the time to actual contact became shorter than the true holding time, and the balance was sufficient for diffusion processes to reach completion over the entire contact area. According to metallographic examination, the joints matched the parent metal in strength when an intercrystalline bond had formed over the larger part of the mating surfaces.

When the bonding temperature was raised to 1 473 K, the ductility and strength of welds in both alloys were reduced. This was attributed to an excessive grain growth in ЭИ602 alloy and to the dissolution of hardening phases in ЭП99 alloy. At a bonding temperature of 1 523 K and a bonding pressure of over 98 MPa, examination revealed cracks along grain boundaries in both alloys. This confirmed the importance of considering the plastic properties in specifying the bonding temperature.

After optimal values for bonding temperature and bonding pressure were determined, tests were conducted to assess the effect of holding time on bond strength. For ЭП99 alloy the minimum holding time was found to be 6 min, and for ЭИ602 alloy, 3 min. It was noted that at a holding time of 0.1 min there was an increase in joint strength. This increase was apparently due to the effect of concurrent strain hardening which is usually cancelled by prolonged heating. The stability of the properties was found to depend on the length of time interval between the instant when the mating surfaces were cleaned and the actual bonding operation. As the time interval was reduced, the stability of the properties improved. On the basis of the tests, the optimal bonding conditions were chosen as follows. For ЭИ602 alloy, T = 1 423-1 448 K, p_b = 24.5-29 MPa, and t = 3 or 4 min. For ЭП99 alloy, T = = 1 423-1 448 K, P_b = 34.3-39 MPa, and t = 6 or 7 min.

BONDING WITH AN AMMONIUM FLUORIDE INTERLAYER. The use of an ammonium fluoride interlayer does not affect qualitatively or quantitatively the relationships that exist between the variables in bonding without an interlayer, but it makes the

quality of welds more consistent and minimizes the effect of the time interval between cleaning and actual bonding to within 24 hours.

BONDING WITH A FUSIBLE INTERLAYER. In a study the interlayer was a Ni-Mn solder containing 30-35% Mn. The remaining elements were in the same concentration as in the alloy. The bonding temperature was set at 1 448 K (which is the melting point of the solder), and the holding time at 6 min. Calculations showed that under the bonding conditions stated a final manganese concentration of 0.4% in the middle of the layer could be achieved with an interlayer 0.1 μm thick, and that a final manganese concentration of 4% could be achieved with an interlayer 1 μm thick. Since a foil thus thin was difficult to make and to handle, it was proposed to obtain the desired thickness for the interlayer by expelling the molten metal of a thicker foil from between the pieces. The objective of the experiment was to determine the optimal bonding pressure and holding time for the case on hand. The studies were carried out on ЭП99 high-temperature alloy. The solder foil was 0.06-0.08 mm thick. The thickness of the interlayer left after expelling the excess molten metal out of the joint was determined by dividing the overall manganese concentration in the diffusion zone (the area under the manganese distribution curve in the joint) into its initial concentration in the interlayer. Distribution curves for manganese and the other elements were obtained with a *Cameca* X-ray microanalyzer which can handle specimens as small as 2 μm across.

As is seen from Fig. 3-24, the thickness of the interlayer generally decreases with an increase in bonding pressure. No advantage could be gained, however, from raising the bonding pressure above 14.7-19.6 MPa, because its effect is drastically reduced. In the range stated, the actual thickness of the interlayer is 1.3 μm, and the Mn concentration is 2.2%, which is very close to the calculated data. As is the case

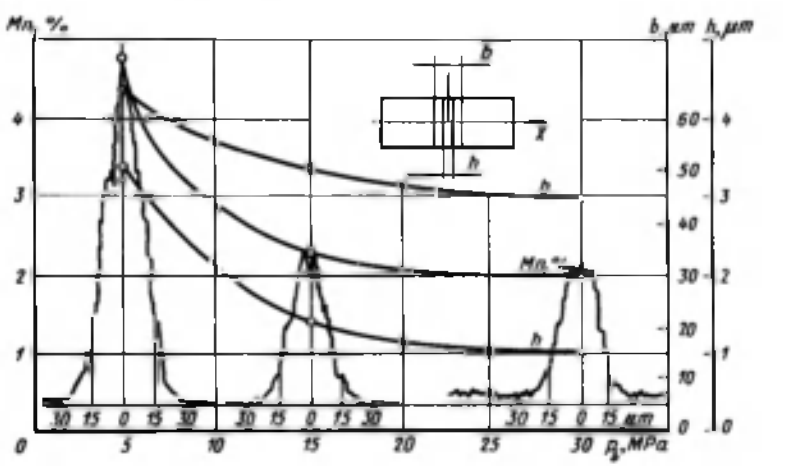


FIG. 3-24 Effect of pressing load on concentration of Mn, interlayer thickness h and diffusion-zone width b for joints in ЭП 99 alloy. T = 1 448 K, t = 6 min

with interlayer thickness, bond strength, too, rises as the bonding pressure is increased. At a bonding pressure of 14.7-19.6 MPa, the joint has practically the same strength as the parent metal.

An increase in holding time leads to a more complete equalization of manganese content throughout the joint (Fig. 3-25). It does not pay, however, to extend the holding time to over 6 min because the rate of content equalization is sharply slowed down.

The distribution of nickel, chromium, and tungsten in the bond zone is shown in Fig. 3-26. As is seen, the elements are distributed uniformly. This is also true of aluminium, molybdenum and titanium. The latter is present in the form of carbides in both the parent metal and the interlayer. The interlayer is seen to carry 19.2% C not present originally. This is an indication that chromium eagerly diffuses into the interlayer from the parent metal carrying 22%C. Tungsten is distributed in the same manner as chromium. The interlayer carries 6% W, and the parent metal, 6.6% W.

In service, high-temperature alloys are heated to 1 173 K or even higher. To assess the effect of a long-term stay (300 hours) at 1 173 K and a short-term stay (16 hours) at 1 373 K on the distribution of the elements in the bond zone, a further study was made. As is seen from Fig. 3-26*b* and *c*, the concentration of chromium is equalized completely, and that of manganese to a noticeable extent. In other words, the chemical homogeneity of high-temperature alloys improves in service.

On the basis of investigations, the following conditions have been recognized as optimal for the diffusion bonding of ЭП99 and ЭИ602 alloys with a fusible interlayer: T = 1 448 K, p_b = 14.7-19.6 MPa, and t = 6 min.

Studies have proved the decisive edge of diffusion bonding with a fusible interlayer over that without an interlayer. Interlayers have been found to stabilize the quality of diffusion-bonded joints, to make them less sensitive to the duration of the time interval between cleaning and the actual joining operation, and to sharply reduce the shrinkage of workpieces due to the plastic deformation produced by bonding. As regards diffusion-bonded joints in alloys carrying an elevated percentage of alloying elements with a high affinity for oxygen (such as aluminium and

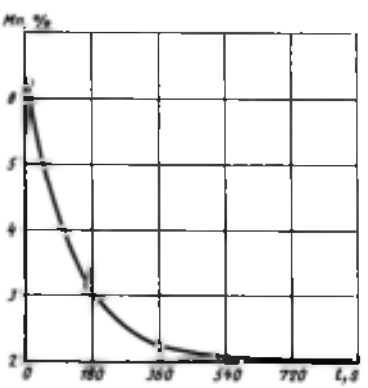


FIG. 3-25 Effect of holding time (duration of pressure) on residual Mn concentration for joints in ЭП 99 alloy. T = 1 448 K, p_b = 14.7 MPa

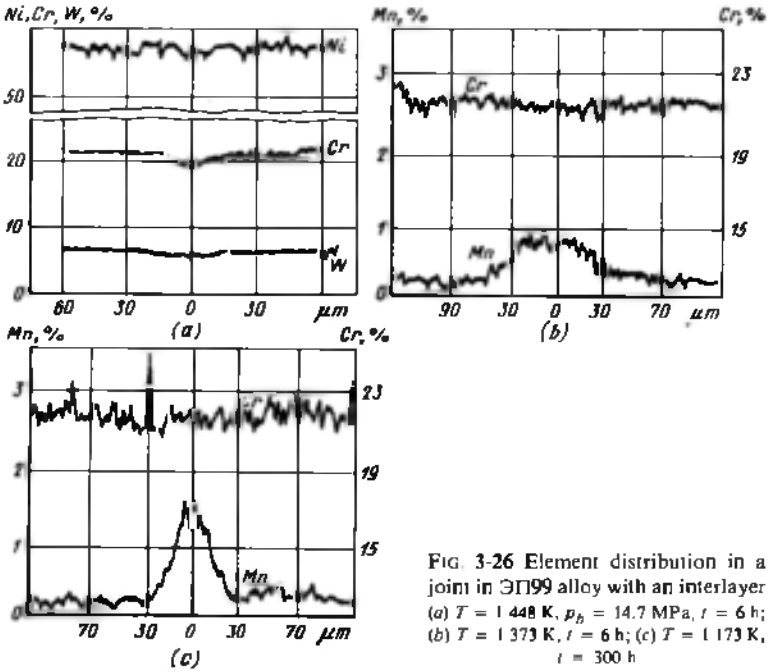


FIG. 3-26 Element distribution in a joint in ЭП99 alloy with an interlayer (*a*) T = 1 448 K, p_b = 14.7 MPa, t = 6 h; (*b*) T = 1 373 K, t = 6 h; (*c*) T = 1 173 K, t = 300 h

titanium), their quality can best be stabilized by active removal of oxide films from the mating surfaces. Also, it will be a good plan to use activated fusible interlayers and active chemical compounds, such as ammonium fluoride, buttered to the mating surfaces. Fusible interlayers are a must in cases where weldments are to meet especially stringent requirements for dimensional accuracy.

The calculated values of dissociation pressure for the oxides of the elements in ЭИ602 alloy in the temperature range 1273-1573 K are given in Fig. 3-27. The partial pressure of oxygen in the process chamber exceeds the dissociation pressure for the oxides of all elements, except nickel. As a consequence, these oxides will not dissociate in the course of diffusion bonding. In fact, one should expect the oxidation of the alloy elements by the oxygen of the gas phase. The highest affinity for oxygen (and the lowest dissociation pressure) is displayed by aluminium, titanium, and silicon. It should be expected, therefore, that at elevated temperatures the oxide films on high-temperature alloys will be enriched by their oxides. If the alloy is not saturated with oxygen, the dissociation pressure will be lower than shown in the plot of Fig. 3-27, and the above conclusion will be valid still more. Similar results have been obtained for ЭП99 alloy. If no air leaks into the vacuum chamber, the oxidation slows down as more of the oxygen is used up. It would cease altogether, if the partial pressure of oxygen in the chamber became equal to the dissociation pressure

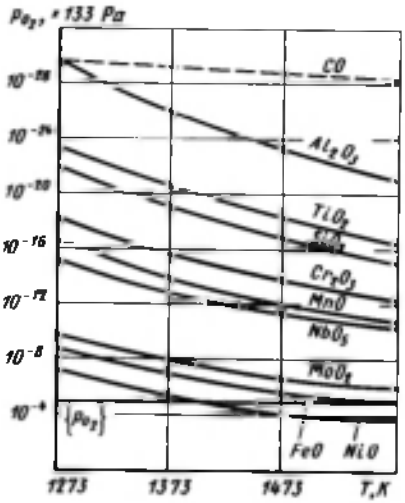


FIG. 3-27 Temperature dependence of dissociation pressure for the oxides of the elements dissolved in ЭИ602 alloy saturated with oxygen. The dissociation pressure of carbon monoxide in the presence of free carbon under a vacuum of 1.3×10^{-2} Pa

of the oxides. Such a state, however, is unattainable from considerations of chemical kinetics as this would require an infinitely long time. To sum up, the vacuum in the welding chamber moderates rather than prevents oxidation, so the use of a higher vacuum would be unwarranted.

The pieces coming in for diffusion bonding are usually machined so that the asperities on the mating surfaces are not higher than 40 μm. Immediately before the bonding operation, the mating surfaces are usually worked with fine-grained emery paper and rinsed with acetone. In cases where use is made of fusible interlayers, cleaning may be done 24 hours before the bonding operation proper. The optimal choice for the fusible interlayer is a Ni-Mn solder (30% Mn) from 0.006 to 0.1 mm thick. Before it is inserted in a joint, the foil should be worked with fine-grained emery paper and rinsed with acetone. If the work design is such that no excess molten metal will be expelled out of the joint (in the case of workpieces with internal passages), the thickness and area of the foil should be reduced. In any case, however, the area of the foil ought not to be less than two-thirds of the contact area. The required amount of molten interlayer metal can be reduced by reducing its thickness. The foil should preferably be tacked to the surface of one of the pieces by stored-energy spot welding, using a suitable resistance welding machine or the usual equipment for diffusion bonding in vacuum.

The recommendations set forth above have been used in making critical parts of an elaborate shape. As compared with brazing, diffusion bonding has substantially improved both the strength and dimensional accuracy of weldments. The diffusion-bonded joints thus made do not fail when bent through an angle of 180°, whereas brazed joints would fail without a marked change in their profile. In tensile tests, diffusion bonded joints frequently fail at the parent metal.

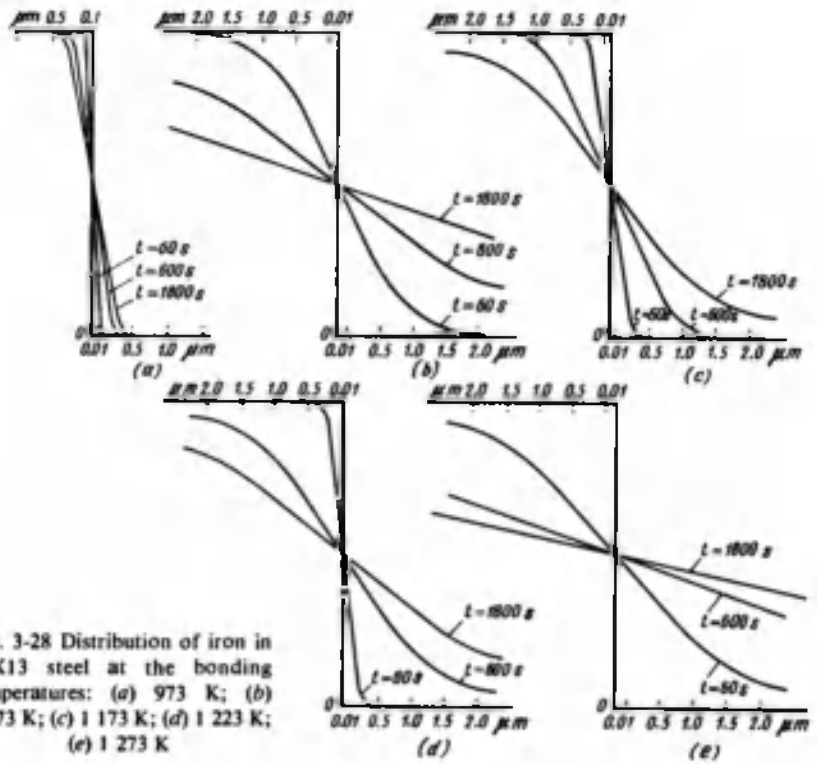


FIG. 3-28 Distribution of iron in 20X13 steel at the bonding temperatures: (*a*) 973 K; (*b*) 1 073 K; (*c*) 1 173 K; (*d*) 1 223 K; (*e*) 1 273 K

Another example of how the bonding variables and procedures can be developed for diffusion bonding is the method based on the concentration of the elements in the diffusion transition zone. Curves based on calculated data appear in Fig. 3-28. As is seen, there is practically no diffusion zone (Fig. 3-28*a*) when the holding time is 30 min and the bonding temperature is 973 K. At a holding time of 30 min and a bonding temperature of 1 073 K (Fig. 3-28 *b*), an insignificant diffusion zone appears. At a bonding temperature of 1 173 K and a holding time of 10 min (Fig. 3-28*c*), an acceptable diffusion zone is formed. A far better chance of making a quality diffusion-bonded joint exists at 1 223 K (Fig. 3-28*d*) and 1 273 K (Fig. 3-28*e*). To sum up, steels, such as 20X13 should preferably be diffusion-bonded at a temperature in the range 1173-1223 K, and the holding time should be 10 min. In this temperature range, the yield strength of the joint has been reported by different authors to be anywhere between 11.7 and 19.6 MPa. The bonding pressure should be taken equal to 15.7 MPa. Experimental data for 20X13 steel are given in Fig. 3-29*a* through *e*. They check well with the calculated optimal bonding variables. In

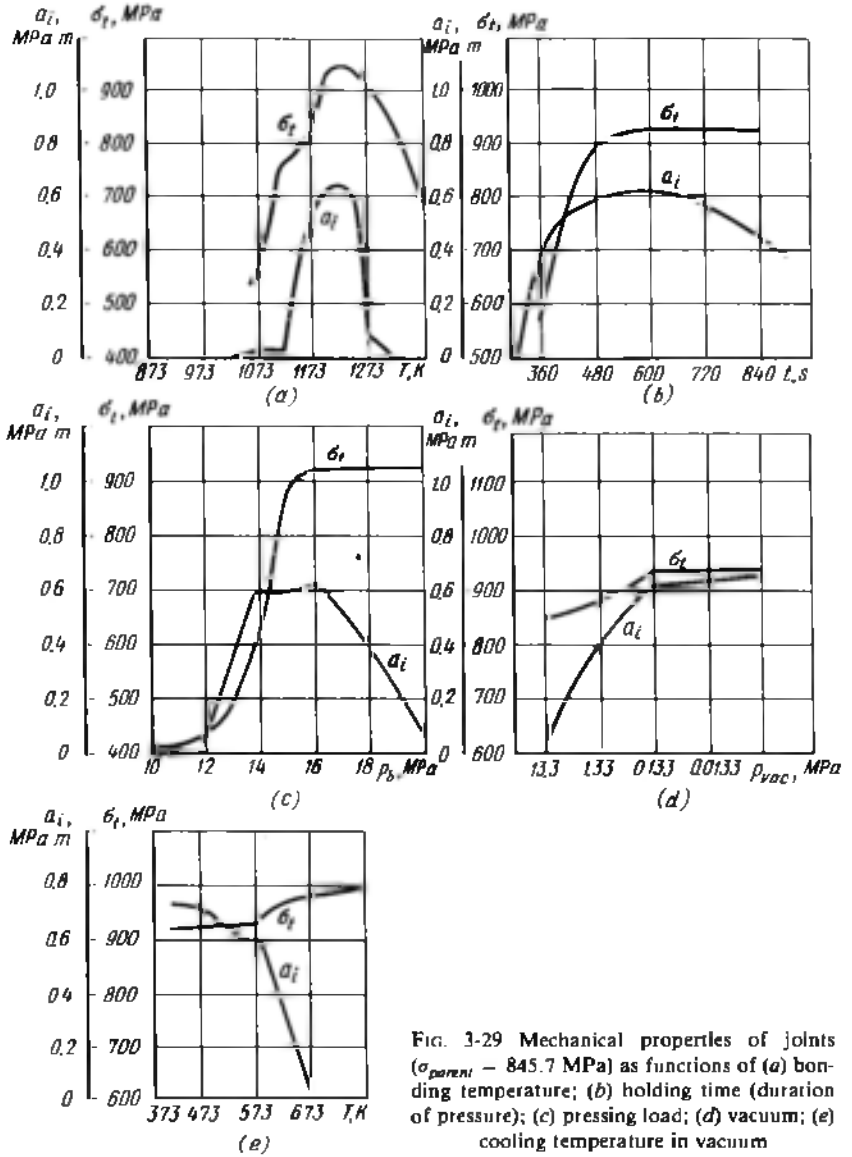


FIG. 3-29 Mechanical properties of joints (σ_{parent} = 845.7 MPa) as functions of (*a*) bonding temperature; (*b*) holding time (duration of pressure); (*c*) pressing load; (*d*) vacuum; (*e*) cooling temperature in vacuum

this respect, variations in impact strength are especially instructive because this property is a structure-sensitive quantity, especially for martensitic steels of which 20X13 steel is an example.

Bibliography

1. KAZAKOV, N. F. (Ed.). *Diffusion Bonding of Metals and Nonmetals.* In: "Transactions of PNILDSV". PNILDSV, Moscow, 1968, 1970, 1971, 1973, 1975, 1976, 1978, and 1979, Nos. 1-9 (in Russian).
2. KAZAKOV, N. F. *Diffusion Bonding.* In: "Welding in Mechanical Engineering", vol. 1. Mashinostroyeniye, Moscow, 1978 (in Russian).
3. KAZAKOV, N. F. *Diffusion Bonding of Materials.* Mashinostroyeniye, Moscow, 1976 (in Russian).
4. KAZAKOV, N. F. *The Formation of the Joint in Diffusion Bonding.* "Svarochnoye proizvodstvo", 1973, No. 9, pp. 48-50 (in Russian).
5. KVASNITSKY, V. F., KOKH, B. A., SOFONOV, A. I. *Diffusion Bonding of High-Temperature Alloys.* Znaniye, Leningrad, 1969 (in Russina).
6. KONYUSHKOV, G. V., ZOTOV, B. M., MERKIN, E. I. *Ferrites and Their Joining to Metals and Ceramics.* Energiya, Moscow, 1979 (in Russian).
7. KONYUSHKOV, G. V., KOPYLOV, Yu. N. *Diffusion Bonding in Electronics.* Ed. by N. F. Kazakov, Energiya, Moscow, 1974 (in Russian).
8. KOCHERGUIN, K. D. *Pressure Welding.* Mashinostroyeniye, Leningrad, 1972 (in Russian).
9. PATON, B. Ye. *The Welding of the Future.* In: "Science and Mankind", vol. 3. Znanlye, Moscow, 1963 (in Russian).
10. KHUDYSHEV, A. F. *Vacuum Diffusion Bonding of Parts for Electron Devices.* Znaniye, Leningrad, 1965 (in Russian).

4

Equipment for Diffusion Bonding

N. F. Kazakov

4.1 General

Engineering to-day heavily depends on the use of refractory metals, high-strength and high-temperature steels and alloys, and also nonferrous metals and alloys which can be joined to produce strong, ductile, and corrosion-resistant welds. To make them by diffusion bonding, one needs equipment specifically designed for the purpose.

At present, a wide range of diffusion-bonding equipment has been developed, differing in design and application. In each particular case, a diffusion bonding unit incorporates all facilities necessary to carry out welding and auxiliary operations.

Bibliography

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2. KAZAKOV, N. F. *Diffusion Bonding.* In: "Welding in Mechanical Engineering", vol. 1. Mashinostroyeniye, Moscow, 1978 (in Russian).
3. KAZAKOV, N. F. *Diffusion Bonding of Materials.* Mashinostroyeniye, Moscow, 1976 (in Russian).
4. KAZAKOV, N. F. *The Formation of the Joint in Diffusion Bonding.* "Svarochnoye proizvodstvo", 1973, No. 9, pp. 48-50 (in Russian).
5. KVASNITSKY, V. F., KOKH, B. A., SOFONOV, A. I. *Diffusion Bonding of High-Temperature Alloys.* Znaniye, Leningrad, 1969 (in Russina).
6. KONYUSHKOV, G. V., ZOTOV, B. M., MERKIN, E. I. *Ferrites and Their Joining to Metals and Ceramics.* Energiya, Moscow, 1979 (in Russian).
7. KONYUSHKOV, G. V., KOPYLOV, Yu. N. *Diffusion Bonding in Electronics.* Ed. by N. F. Kazakov, Energiya, Moscow, 1974 (in Russian).
8. KOCHERGUIN, K. D. *Pressure Welding.* Mashinostroyeniye, Leningrad, 1972 (in Russian).
9. PATON, B. Ye. *The Welding of the Future.* In: "Science and Mankind", vol. 3. Znanlye, Moscow, 1963 (in Russian).
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Equipment for Diffusion Bonding

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4.1 General

Engineering to-day heavily depends on the use of refractory metals, high-strength and high-temperature steels and alloys, and also nonferrous metals and alloys which can be joined to produce strong, ductile, and corrosion-resistant welds. To make them by diffusion bonding, one needs equipment specifically designed for the purpose.

At present, a wide range of diffusion-bonding equipment has been developed, differing in design and application. In each particular case, a diffusion bonding unit incorporates all facilities necessary to carry out welding and auxiliary operations.

A typical diffusion bonding unit consists of two subunits, electromechanical and power-supply. The function of the electromechanical subunit is to seal and evacuate the process chamber, to set up, move and weld the workpieces, to perform all auxiliary operations, and to control all the steps. Accordingly, it includes a vacuum chamber, a vacuum pumping system, a system to transmit motion to the workpieces, an observation system, and auxiliaries to monitor and control the temperature of workpieces at the contact zone and the vacuum in the chamber, and also to control the electrical devices used. The power subunit is essentially an assembly of heating sources. These may use induction, radiation, lasers, an electron beam, a glow discharge, a flow of electric current, and their combinations.

A major distinction of diffusion bonding is the use of vacuum. Even at a pressure of 133 Pa the impurity content of the atmosphere in the process chamber is lower than that of commercially pure argon. Apart from its shielding action, vacuum provides for a faster and more complete degassing of the materials being joined and the removal of oxides, impurities and contaminants from both the surface and the bulk of the materials. In view of its importance, it is worthwhile to discuss vacuum and vacuum techniques in more detail.

4.2 Vacuum Techniques

BASIC THEORY. In an engineering sense, the term "vacuum" may refer to the range of pressures extending from atmospheric down to the lowest that can be produced at the present state of the art. Arbitrarily, the entire range may be divided into five subranges as shown in Table 4-1.

Industrial units usually operate at low and high vacuum, that is, at a pressure of 1.3×10^{-1}-1.3×10^{-4} Pa. For a proper design of vacuum plant it is essential to know the behaviour of gases in that pressure range.

As will be recalled, molecules (atoms) in a solid, liquid or gas are continuously moving at random. This movement is responsible for a number of traits essential to vacuum techniques.

1. The property of gases to take up all of the volume available to them is utilized for their removal from vessels by, say, mechanical pumps.

TABLE 4-1

Vacuum	Pressure, Pa	Application
Low	1.3×10^{-1}	Gas-filled tubes
High	1.3×10^{-1}-1.3×10^{-4}	Electric lamps
Very high	1.3×10^{-4}-1.3×10^{-7}	Transmitting tubes
Ultra-high	1.3×10^{-7}-1.3×10^{-10}	X-ray tubes, equipment for producing extremely pure gases
Cosmic	1.3×10^{-10}-1.3×10^{-12}	In nebulae and outer space

2. The property of gases to interpenetrate one another (interdiffusion) is utilized in diffusion vacuum pumps which are capable of building up extremely low pressures.

3. The thermal motion of air particles is responsible for in-leakage in vacuum systems, that is, ingress of atmospheric air inside a vacuum system around poorly sealed or unsealed joints.

The pressure of a gas may be defined as the sum of the momenta imparted by gas molecules as a result of their thermal motion to every square centimetre of the confining vessel's walls per second. In moving around, gas molecules not only hit the vessel's walls, but also collide with one another. Because of this, molecules move in a zig-zag pattern in which the straight portions are those along which molecules move between collisions (free paths). The points where these paths meet correspond to the instants at which molecules collide. Since thermal motion is random, the free paths cannot be all of the same length. Fortunately, they obey a distribution law, so we may use the concept of the mean free path, symbolized by the Greek letter λ (lambda).

The mean free path, λ, bears a well-defined relationship to the mean number of collisions per second, Z:

$$\lambda = V_a/Z$$

Hence, if V_a is held constant, the mean free path of gas molecules is inversely proportional to the mean number of collisions per second. By the kinetic theory of gases, the mean free path of molecules is inversely proportional to the molecular concentration (molecular number density), N, of the gas. Therefore, if the temperature is held constant, the mean free path is given by

$$\lambda = k/p$$

or

$$\lambda p = k$$

where k is a coefficient equal to the mean free path of molecules of a given gas at a pressure equal to unity. A more complete definition of the mean free path leads to the following expression

$$\lambda = \frac{1}{\sqrt{2} N_1 \pi \sigma^2 (1 + C/T)} \tag{4-1}$$

where N_1 is the molecular concentration (number density) of the gas, σ is the mean diameter of molecules, T is the absolute temperature of the gas, and C is the constant of that gas.

In Eq. (4-1), apart from the inverse proportionality between λ and N_1, it is important to note the dependence of the mean free path on the cross-sectional area of molecules given by the product $\pi\sigma^2$. The term $(1 + C/T)$ has been found experimentally to account for the temperature dependence of the mean free path.

If we designate the mean free path at a temperature of 273 K as λ_{273}, and that at a temperature T as λ_T, then

$$\lambda_T = \lambda_{273} \frac{(273 + C)T}{(T + C)273} \tag{4-2}$$

Using Eq. (4-2), we can find the mean free path at a given temperature if we know its value at some other temperature. Examples of computed values of λ for some gases at a temperature of 273 K are given in Table 4-2.

TABLE 4-2

Gas	λ at 273 K and 1325 Pa, cm $\times 10^{-6}$	C
N_2	5.99	102.7
Ar	6.35	147.8
H_2	11.23	82.8
O_2	6.47	136.0
Air	6.08	124.0

As an example, for air at 293 K we obtain

$$\lambda_{293} = \frac{6.08 \times 10^{-6}(273 + 124) \times 293}{(293 + 124) \times 273} = 6.21 \times 10^{-6} \text{ cm} \tag{4-3}$$

On the basis of Eq. (4-2) and Table 4-2, also for air at 293 K the product of mean free path and pressure is

$$\lambda_p = \text{approx. } 0.665 \text{ cm Pa}$$

Using this relation, we can compute the value of λ for air at any pressure

$$\lambda = 0.665/p \text{ (cm)}$$

The mean free path at temperatures other than given in the table can be found from a simple relation

$$\lambda_m = \lambda(T_2/T_1)$$

where T_1 is the room temperature (293 K) and T_2 is the actual temperature.

The dependence of the mean free path for air at 293 K and the molecular number density on pressure is given in Table 4-3.

As follows from Table 4-3, the popular concept of vacuum as an emptiness is wrong, because even at a pressure of 1.3×10^{-6} Pa which can only be produced by high-performance high-vacuum vapour jet pumps, every cubic centimetre contains over three thousand million gas molecules.

The key properties of gases and the events occurring in the container can be defined according to the ratio of the mean free path, λ, and the characteristic dimension, D, of the confining vessel. The state at which $\lambda \gg D$ is referred to as high vacuum. At $\lambda \ll D$, we have low vacuum. The state in which $\lambda \lesssim D$ is sometimes called intermediate vacuum.

TABLE 4-3

Air pressure, Pa	λ, cm	Molecular number density at 293 K
1325	6.21×10^{-6}	2.5×10^{19}
133	4.72×10^{-3}	3.3×10^{16}
13.3	4.72×10^{-2}	3.3×10^{15}
1.3	4.72×10^{-1}	3.3×10^{14}
1.3×10^{-1}	4.72	3.3×10^{13}
1.3×10^{-2}	4.72×10^{1}	3.3×10^{12}
1.3×10^{-3}	4.72×10^{2}	3.3×10^{11}
1.3×10^{-4}	4.72×10^{3}	3.3×10^{10}
1.3×10^{-5}	4.72×10^{4}	3.3×10^{9}
1.3×10^{-6}	4.72×10^{5}	3.3×10^{8}
1.3×10^{-7}	4.72×10^{6}	3.3×10^{7}

A vacuum system refers to an assemblage of devices which produce and measure the degree of rarefaction, and pipework which interconnects them. It is designed on the assumption that the volume of gas, Q, passing through any section of the system per unit time (volumetric flow rate) remains constant. Then the conductance of the pipework can be written as

$$C_{pw} = Q/(p_1 - p_2)$$

where p_1 is the inlet pressure, p_2 is the outlet pressure, and Q is the volumetric flow rate, Pa m^3 s^{-1}.

The amount of gas, Q, entering the pump per unit time at the inlet pressure is known as the pump or intrinsic speed, S_p. It is defined as

$$S_p = Q/p_1$$

The amount of gas, Q, entering the pipework of conductance, C_{pw}, per unit time per unit pressure, from the vessel being pumped, is called the speed of exhaust, S_{ex}. It is an easy matter to show that

$$1/S_{ex} = 1/C_{pw} + 1/S_p \tag{4-4}$$

As follows from Eq. (4-4), the speed of exhaust and the pump speed, which must be known before an adequate pump size can be chosen, are connected by the conductance of the pipework. This latter quantity varies according to the flow regime of the gas, the kind of gas, the geometry of the pipework, and some other factors. Let us see how the flow regime of a gas through a given pipework varies with pressure.

At high pressures and high gas velocity, the gas particles move haphazardly. Quite aptly, this regime is called turbulent flow. As the pressure and velocity of the gas are brought down, the turbulent flow gives way to an ordered, streamline flow. In this regime the velocity of the gas gradually increases on moving away from the

pipe walls towards the centre. Now the controlling quantity is the viscosity of the gas, so this regime is called viscous flow [2, 4, 6, 7]. As the pressure is brought down still more, the amount of gas decreases, and the mean free path increases. When the mean free path becomes equal to or greater than the diameter of the vessel or pipework, molecules collide less frequently than they hit the walls (the probability of molecules colliding is said to be lower than the probability of hitting the walls). Now the molecules travel independently of one another, and the regime is known as molecular flow.

It should be stressed that there is no abrupt change from one flow pattern to another. Instead, one regime changes to another through what is called a transitional flow or region.

To begin with, let us define the limits of viscous flow in pipework. As already noted, the transition from turbulent to viscous flow is marked by an increasingly ordered movement of molecules. The criterion of this transition is usually expressed in terms of the Reynolds number, a dimensionless group written as

$$\mathrm{Re} = dv\rho/\eta \tag{4-5}$$

where d is the diameter of the pipework in cm, v is the velocity of the gas flow, ρ is the density of the gas, and η is the viscosity of the gas. It has been proved experimentally that viscous flow can exist at $\mathrm{Re} < 1\,200$. On expressing the velocity and density of the gas in Eq. (4-5) in terms of the flow rate of the gas, and also the pressure, simple manipulations give another expression for the Reynolds number

$$\mathrm{Re} = Q/89d$$

$$Q \leqslant 106\,800\,d$$

Thus, the upper limit (in terms of pressure) for viscous flow to exist is set by $Q \leqslant 1 \times 10^5$. In this regime, the conductance of a pipework for air at 293 K is given by

$$C_{pw} = 0.182pd/l \tag{4-6}$$

where C_{pw} is the conductance of the pipework in $1\ s^{-1}$, d is the diameter of the pipework in cm, l is the length of the pipework in cm, and p is the mean pressure in the pipework in Pa.

The conductance of pipework for other gases relative to that for air in the case of viscous flow and a temperature of 293 K is given in Table 4-4.

TABLE 4-4

Medium	C_{gas}/C_{air}
Hydrogen	2.1
Helium	0.93
Water vapour	1.9
Neon	0.54
Nitrogen	1.04

As the pressure is brought down and the mean free path of molecules becomes comparable with the diameter of the pipework, Eq. (4-6) ceases to be exact. It is assumed that Eq. (4-6) gives the conductance in the viscous regime if the error does not exceed 10%. Accordingly, the lower limit of the viscous region is set by

$$pd > 6.5 \times 10^{-2} \text{ Pa m}$$

The molecular regime exists at high vacuum when the mean free path of molecules exceeds the diameter of the pipework. The lower limit of the molecular region is set by

$$pd < 0.19 \times 10^{-2} \text{ Pa m}$$

Now the conductance of the pipework is independent of pressure. For a round long pipework in which the molecular regime exists, the conductance at 293 K is given by

$$C_{pw} = 12.1\, d^3/l$$

For a short pipework account must be taken of the throttling effect produced as a gas passes from a vessel of a larger cross section, $1/C_p$, into a vessel of a smaller cross section, $1/C_{th}$. The total conductance can be found from

$$1/C_{pw} = 1/C_p + 1/C_{th} \tag{4-7}$$

where C_p is the conductance of the pipes proper, and C_{th} is the conductance of the throttle (orifice plate).

In the case of molecular flow, the conductance of the orifice plate is given by

$$C_{th} = \frac{11.6A}{1 - A/A_0}$$

where A is the area of the orifice, and A_0 is the cross-sectional area of the vessel from which the orifice lets out the gas.

For a circular pipework,

$$C_{th} = \frac{9.11d^2}{(1 - d/D)^2}$$

where d is the diameter of the pipes or orifice, and D is the diameter of the confining vessel supplying the gas.

In view of the foregoing, we may re-write Eq. (4-7) for short pipework in the case of molecular flow as

$$C = \frac{12.1d^3/l}{1 + 1.33d(1 - d^2/D^2)/l} = 12.1\alpha d^3/l$$

where α is the throttling factor.

For an annular pipework,

$$C = 12.1 \frac{(d_1 - d_2)(d_1 + d_2)}{L} K$$

where the value of K depends on the magnitude of the ratio d_2/d_1. Several values of K are given in Table 4-5.

TABLE 4-5

d_2/d_1	0	0.259	0.500	0.707	0.866	0.966
K	1	1.072	1.154	1.254	1.43	1.675

In all cases involving a short pipework, account must be taken of the throttling effect produced in changing from a larger to a smaller diameter.

In designing a vacuum system it is important to consider the transitional regime when the flow is partly viscous and partly molecular or, which is the same, molecules both collide with one another and with the walls of the confining vessel. As already stated, the upper limit for the transitional region is set at $pd < 6.5 \times 10^{-2}$ Pa m, and the lower limit at $pd > 0.19 \times 10^{-2}$ Pa m. Within these limits, the Knudsen formula holds, which for air at 293 K has the form

$$C = 12.1(d^3/l)\frac{1 + 0.271dp + 0.00479(dp)^2}{1 + 0.316dp}$$

where p is the mean pressure in the pipework in Pa.

The above equation may be simplified as follows

$$C = 12.1(d^3/l)K$$

To sum up, a generalized equation for the conductance of circular pipework of any length under conditions of molecular, transitional and viscous flow takes the form

$$C = 12.1K\alpha d^3/l \tag{4-8}$$

The material set forth so far can only be used in the simplest cases. Many components of a vacuum system can hardly be treated theoretically, so their conductances have to be determined by experiment.

The pump-down time from atmospheric pressure can be found by the following equation

$$t = 2.3(V/S_{ex})\log_{10}(p_1/p_2)$$

where V is the volume of the vacuum vessel in litres, S_{ex} is the speed of exhaust in l s^{-1}, p_1 is the pressure in the vessel at the start of the pump-down, and p_2 is the pressure at the end of the pump-down.

In most cases involving forevacuum lines, the speed of exhaust from the chamber of a diffusion-bonding unit is very close to the pump (intrinsic) speed. Therefore, in preliminary calculations it is safe to take $S_{ex} = S_p$. Sometimes, the figures thus found should be verified by analysis, which is especially important in the case of long, small-diameter pipelines.

In getting down to the design of a vacuum system, it is necessary above all to define its application, the ultimate pressure desired, the required pump-down time or the throughput of the system. Once these quantities are determined, the designer chooses:

— the layout of the lines, the throughput of the individual portions, and the amount of outgassing and in-leakage in the system;

— the basic pumping facilities, baffles, traps, getters, etc.;

— the materials of construction to be used (metals, glass, ceramics, rubber, etc.) and the processes for joining them (brazing, welding, etc.);

— the types of vacuum valves, leaks, motion seals, and demountable joints;

— the types of vacuum gauges used, the points of connection, and the manner of connection to the vacuum system.

Extremely diverse as they are, vacuum-system components are expected to meet a number of common requirements which may be summed up as follows.

(1) Vacuum tightness. To satisfy this requirement, vacuum engineering uses a particular range of gas-tight materials and high vacuum tightness is specified for welded, brazed and demountable joints.

(2) Chemical stability, especially when a vacuum system is intended to handle corrosive or poisonous substances. Because of this, vacuum engineering uses a wide range of corrosion-resistant steels and alloys.

(3) Thorough treatment of inside surfaces for components operating at high vacuum (a pressure of less than 1.3×10^{-4} Pa).

(4) Ample mechanical and thermal strength and constancy of geometry. These requirements are especially important where vacuum-system components are to operate at elevated pressures inside and, sometimes, outside the chamber, and also at elevated and cryogenic temperatures. Vacuum units are preferably made with thin walls, so the constancy of geometry is an important consideration in vacuum work.

(5) The ability to operate at a high electric potential, in a strong magnetic or electric field, and under other unusual physical conditions. To assure this ability, vacuum-system components are fabricated from materials especially adapted to a particular environment (dielectrics, nonmagnetic alloys, etc.) [7, 12].

Vacuum-system components are expected to meet the above requirements singly or in combinations, depending on their application. Let us consider them in more detail.

VACUUM TIGHTNESS. Even a minute amount of gas finding its way into the vacuum chamber may drastically affect the vacuum and knock the unit out of service [2, 4, 5, 7, 10, 12, 14]. In this respect vacuum systems differ from any other equipment, including high-pressure facilities.

Example. If, from an ordinary compressed oxygen cylinder with a capacity of 40 l and containing oxygen compressed to 49×10^5 Pa, we withdraw an amount of gas equivalent to 1 cm^3 at a pressure of 98 Pa, the pressure in the cylinder will change by a mere 1/6 000 000 (by 0.000 017%). In most practical cases, this reduction in pressure may be ignored.

The situation is different if the same amount of gas is allowed to enter a similar cylinder but evacuated to a pressure of 1.3×10^{-4} Pa. Prior to admission (or in-leakage) the evacuated cylinder holds an amount of gas which at a pressure of 98 Pa takes up a volume equal to

$$40\,000 \times 10^{-6} \div 760 = 1/19\,000 \text{ cm}^3$$

Hence, 1 cm^3 of gas is 19 000 times that amount, and the pressure in the cylinder will increase 19 000 times as a result of admission (or in-leakage).

Because of the requirement for tightness, utmost care must be exercised in making joints and seals in a vacuum system. It is around joints (permanent or demountable) that minute cracks are likely to develop. Special emphasis is placed on demountable joints between external components, which in vacuum engineering are sealed with rubber, plastic, or metal gaskets. In vacuum work, it would be unreliable to fill asperities or discontinuities with a paste, cement or paint, or to caulk joints in pipelines and at valves with yarn or a similar material. The only reliable way to make such joints vacuum-tight is through the use of welding, brazing, demountable mechanical joints sealed with smooth metal or elastic gaskets made of materials with a minimal outgassing in vacuum, or ground joints sealed with special grades of grease. Sometimes, components made of plastics may be joined with cements or solvents applied under pressure.

OUTGASSING FROM THE COMPONENTS OF A VACUUM SYSTEM. In addition to in-leakage, the pressure in an evacuated chamber may rise because of the outgassing from its walls. Even if the vapour pressure of the chamber material at a given temperature is negligible and the chamber has been thoroughly cleaned, the outgassing from its walls will be rather noticeable. One of its causes is the presence of gases and vapours in the bulk of the materials which go to make a vacuum system. This is especially true of welded and brazed joints, rubber and plastic gaskets, ceramic through-feeds, and motion seals. Rubber, most plastics and nearly all kinds of ceramics can take up certain amounts of moisture. Another cause for outgassing is the adsorption of gases and liquids onto the surfaces of vacuum-system components under a vacuum. Obviously, a rough inner wall will have a larger effective surface area and a greater capability to adsorb gases. Complete separation of the adsorbed gases from surfaces can only take place at temperatures well above the normal boiling points of the adsorbed substances. Therefore, vacuum-system components are to meet a number of additional requirements:

(1) There must be as few internal welded and brazed joints as practicable.

(2) Inner joints should be welded by processes which produce the least quantity of porous weld metal. These may be gas-shielded arc welding without a filler metal, submerged-arc welding, or argon-arc welding. With these welding processes, oxidation is prevented so there is no scale to be removed. The welds thus made are free from cracks and blowholes.

(3) Any traces of flux should be removed from both brazed and welded joints, the joints must be pickled and carefully rinsed.

(4) Inner surfaces must be machined to a finish such that surface asperities are no more than 0.63 μm high.

(5) Use should be made of anti-corrosion metals.

(6) The demountable joints to be used must be of a design which permits baking at 573-673 K and more for several hours.

The requirements in (4), (5), and (6) are a must when a system is pumped down to a pressure of less than 1.3×10^{-4} Pa.

A typical vacuum system contains suitably designed traps intensively cooled on their working surfaces and intended to remove or combine the vapours or gases condensing in vacuum. They are built along the same lines as other vacuum-system components.

MECHANICAL LOAD AND STRENGTH OF ENCLOSURES. At ordinary pressure vacuum chambers have to resist no other mechanical loads except the outside pressure. Therefore they are designed for a uniformly distributed external load of 1 kg cm^{-2} of their wall surface area. This negligible pressure permits them to be built with thin walls but rigid in shape, especially in the case of a more or less large chamber fitted with a dished, a box-shaped or conical lid, a substantial bottom, and connecting flanges. Flat and rectangular walls, bottoms and lids should be avoided in vacuum chambers, except where no other configuration can serve the purpose. In any type of vacuum equipment preference should be given to cylindrical shapes with shells built up of standard seamless drawn or rolled tubing in the case of smaller chambers or tubing rolled of sheet material in the case of larger chambers. For small chambers operating at normal pressure the wall thickness is usually specified from engineering considerations rather than from strength analysis. The walls should have a thickness sufficient for reliable and inexpensive welding, brazing, or mechanical attachment. For steel chambers operating without elevated pressure the recommended wall thicknesses, in mm, are given in Table 4-6.

GASES DESORBED FROM THE CHAMBER WALLS. As will be recalled, the readily condensing gases (water vapour, hydrocarbons, etc.) when adsorbed onto a solid surface form a layer several molecules thick, whereas the gases hydrogen, oxygen, nitrogen and some others usually produce a monomolecular layer. When a vacuum chamber is evacuated, the adsorbed gases are desorbed, and the pressure in the chamber goes up. The outer layers of molecules are relatively easy to evacuate, whereas the monolayers do so at a slower rate. The rate of desorption can be raised by heating. The amount of gases that will be desorbed from the chamber walls

TABLE 4-6

Dia of cylindrical shell	Wall thickness	Dished lid	Cone lid	Water jacket
200	2.0	2.0	2.0	1.5
400	2.5	3.0	2.5	1.5
500	3.5	3.5	3.0	2.0
600	4.0	4.0	3.0	2.0
700	4.5	4.5	3.5	2.5
800	4.5	4.5	4.0	2.5

depends on their condition and surface finish. Even for a well-machined metal the true surface area is greater than the geometrical one. For platinum foil their ratio is 2.2, for rolled nickel anywhere between 3.5 and 3.8, and for polished nickel anywhere between 7.5 and 9.7. Obviously, the better the surface finish, the smaller the ratio and the smaller the amount of gases that can be adsorbed on a unit surface area. In putting together the components of a vacuum system it is important to rub them with petrol or acetone, then to rinse them with alcohol — in this way outgassing during the pump-down can be greatly reduced.

In diffusion bonding which proceeds at relatively high temperatures, outgassing is more pronounced, and the pressure in the chamber may rise sharply. In fact it becomes a controlling factor where high-vacuum diffusion bonding units are involved, especially those operating at a pressure of 1.3×10^{-4}-1.3×10^{-7} Pa.

In calculating a vacuum system, gas desorption can be predicted through the use of experimental data for outgassing from metal surfaces found by several investigators and presented in Tables 4-7 through 4-10. In the tables, the values are reduced to normal conditions.

TABLE 4-7

Metal	Rate of outgassing, $\times 10^5$ cm^3 cm^{-2} h^{-1}, after evacuation for 2 h
Copper	5.67
Nickel	2.36-2.84
Iron	0.944
Aluminium	1.42-3.3
Molybdenum	1.65-3.08
Tantalum	2.131
Tungsten	0.472

As is seen from Table 4-10, surface pretreatment has a marked effect on the amount of outgassing. For example, outgassing from untreated copper after 5 hours of pumping is by two orders of magnitude higher than it is from copper pickled and degreased with petrol or acetone. The rates of outgassing from stainless steel obtained by several investigators are nearly the same. According to A. L. Mints and

TABLE 4-8

Steel	Rate of outgassing, cm^3 cm^{-2} h^{-1}, after pumping out for the time stated, h					
	1	2	4	6	8	10
Low-carbon	1.5×10^{-3}	8.5×10^{-4}	2.2×10^{-4}	2.6×10^{-4}	1.9×10^{-4}	1.5×10^{-4}
Corrosion-resistant	8.5×10^{-4}	3.8×10^{-4}	2.1×10^{-4}	1.5×10^{-4}	1.1×10^{-4}	9.0×10^{-5}

TABLE 4-9

Metal	Gas content of metal						
	Oxygen		Nitrogen		Hydrogen		Total
	%	$\frac{cm^3}{100\ g}$	%	$\frac{cm^3}{100\ g}$	%	$\frac{cm^3}{100\ g}$	$\frac{cm^3}{100\ g}$
Nickel							
ordinary	—	—	—	—	—	180	—
electrolytic	—	—	—	—	—	164	—
in bars	—	—	—	—	—	—	482
carbonyl	—	—	—	—	—	—	113
Copper	—	—	—	—	—	—	7.97
	—	—	—	—	—	—	0.7-2.7
	—	—	—	—	—	—	0.5-0.9
oxygen-free	0.00045	0.315	—	—	0.00012	1.34	—
vacuum-smelted	0.00004	0.028	—	—	0.00001	0.112	—
Aluminium	—	—	—	—	—	—	4-17
	—	—	—	—	—	—	0.4-1.3
Titanium	0.01-0.4	7-280	—	—	0.02-0.71	224-8230	—
	0.12	84	0.03	24	0.0045	50.4	158.4
Molybdenum							
raw	0.006-0.009	4.2-6.3	0.001-0.003	0.8-2.4	0.001 max	11.2	16.2-19.9
vacuum treated:							
3.9-5.3 Pa	0.004-0.005	2.8-3.5	—	—	—	—	—
0.67 Pa	0.002-0.0035	1.4-2.45	0.0001	0.08	—	—	1.48-2.53
1.3×10^{-2} Pa	0.001	0.7	—	—	—	—	0.7

TABLE 4-10

Material and pretreatment	Rate of outgassing after pumping for 1 h, $cm^3\ cm^{-2}\ h^{-1}$	Rate of outgassing after pumping for 5 h, $cm^3\ cm^{-2}\ h^{-1}$
Copper, pickled, degreased with petrol and acetone	4×10^{-5}	3×10^{-6}
Brass, pickled, degreased with petrol and acetone	7×10^{-5}	6×10^{-6}
Duralumin, pickled, degreased with petrol and acetone	1.7×10^{-4}	2×10^{-5}
Copper, degreased with petrol and acetone	2.2×10^{-4}	4×10^{-5}
Stainless steel, untreated	6×10^{-4}	7×10^{-5}
Copper, untreated	7×10^{-4}	2×10^{-4}
Brass, degreased with petrol and acetone	8×10^{-4}	3×10^{-4}
Duralumin, degreased with petrol and acetone	1×10^{-3}	5×10^{-4}
Duralumin, untreated	2×10^{-3}	7×10^{-4}
Weld on brass tube	2×10^{-2}	8×10^{-2}
Weld on copper tube	2×10^{-1}	3×10^{-2}

N. M. Kristi, outgassing from carbon steels may be by two or three orders of magnitude higher than it is from copper [10]. Heating speeds up outgassing in vacuum. Therefore, if desorption is not to impair the required high vacuum, it is strongly recommended to preheat glass to 673-773 K, and graphite and molybdenum to 773-873 K. Studies concerned with outgassing from Armco iron have shown that at 923 K outgassing is slow, whereas at 1 073 K the metal gives up nearly all of its gases.

In conclusion, it should be noted that outgassing from the metal surfaces in a vacuum system ought not to be neglected, but the absolute values of outgassing under ordinary service conditions are not a decisive factor in the choice of vacuum equipment.

Now, using Table 4-6 or 4-10, let us calculate outgassing from the walls of a vacuum chamber fabricated from corrosion-resistant steel. If the chamber diameter is 1 600 mm, its height is 2 000 mm, and the operating vacuum is 1×10^{-2} Pa, the rate of outgassing will be

$$S_{out} = \frac{6 \times 10^{-4} \times 10^5 \times 760}{1 \times 10^{-4} \times 3600 \times 10^3} = 127\ l\ s^{-1}$$

For normal operation at an outgassing rate like this, use may be made of vacuum pumps with an intrinsic speed of not less than 8 000 $l\ s^{-1}$ in the pressure range 1×10^{-2}-1×10^{-3} Pa. If we assume that the speed of exhaust under conditions of molecular flow is about half the pump speed (that is, 4 000 $l\ s^{-1}$), it is obvious that outgassing at a rate of 127 $l\ s^{-1}$ is not a factor in the choice of a vacuum pump. It should be remembered that in order to obtain a high vacuum in the chamber (1×10^{-5} Pa and better), it must be baked out to 623-673 K for a long time so as to

remove the gases dissolved in the metal. The amount of gas dissolved is about 20 times that adsorbed on the surface.

IN-LEAKAGE. A gas leaking into a vacuum system may react with the workpieces. Therefore in-leakage is a major characteristic affecting the quality of a vacuum chamber.

The in-leakage, H, into a vacuum chamber is given by

$$H = V\Delta p/\Delta t$$

where Δp is the pressure in the chamber, V is the volume of the chamber.

In-leakage is determined twice: at the maker's and by the user. It is usual to determine both outgassing and in-leakage (this quantity is termed the total gas load). In determining in-leakage, the chamber should be pumped out for a long time so as to bring outgassing to a minimum. The absolute value of in-leakage depends on the size of the chamber, operating pressure, throughput of the vacuum system, and the properties of the materials involved. For a unit using a mercury-vapour vacuum pump with an intrinsic speed of 5 l s^{-1} and operating pressure of 1.3×10^{-1} Pa the in-leakage should be not more than 6.5×10^{-10} Pa m^3 s^{-1}. For very large industrial pumps with an intrinsic speed of up to 30 000 l s^{-1} and an operating pressure of 1.3×10^{-3} Pa the in-leakage may be as high as 3.9×10^{-3} Pa m^3 s^{-1}. As experience with the design and manufacture of vacuum units with a chamber up to 12 m^3 in size shows, for equipment operating at pressures 1.3×10^{-1}-1.3×10^{-2} Pa, that the in-leakage ought not to exceed $1.3 \times \times 10^{-4}$ Pa $m^3 s^{-1}$. The figures quoted apply to empty vacuum chambers and characterize their vacuum tightness.

In-leakage is an important indicator of how efficient a vacuum unit is in service. This is why all joints in a vacuum system must be carefully tested both at the maker's and by the user. Should the test show that the in-leakage is grossly in excess of the figure determined by the maker, it is important to run a check on all the joints and gaskets, and remove the cause. It is only then that the equipment may be used for diffusion bonding.

DETERMINING THE REQUIRED SPEED OF EXHAUST. Once the basic causes of vacuum impairment are determined, the system proper may be calculated. The rate of outgassing from several selected metals is given in Tables 4-7 through 4-10. It is also important to know the total amount outgassed, which is a function of time and of temperature. This quantity will also be affected by the rate of heating, but the latter is usually chosen from process considerations.

In calculations it is important to allow for variations in outgassing with time. This is done by applying correction factors. The total amount of gas to be exhausted (the total gas load) is given by

$$Q = 10q_1Gn/t + q_2A + H$$

where q_1 is the outgassing from the workpieces, G is the mass of the workpieces, q_2 is the outgassing from unit area of the chamber walls, A is the area of the walls and internals, H is the in-leakage in μl s^{-1}, t is the cycle time in s, and n is a coefficient taking care of variations in outgassing with time.

The required speed of exhaust can then be found by the following equation

$$S_{ex,req} = 10Gq_1n/tp + q_2A/p + H/p$$

where p is the pressure, in Pa, at which the process is conducted.

4.3 Bonding Chambers

PRINCIPAL REQUIREMENTS. The bonding chamber is a key and critical component of a diffusion bonding unit. The chamber is fitted with access holes through which workpieces are moved into and out of the chamber, attachment fixtures for the heat source (which may be an inductor, a resistance element, or some other device depending on the power supply used), observation windows or ports, motion seals, pressure sensors, electric lead-ins, leaks, etc. It also has ports for connection of a pumping system and of a mechanism transferring the pressing load (bonding pressure) on to the workpieces. All openings in the chamber have thick-walled flanges with well-machined surfaces for proper sealing.

A bonding vacuum chamber is expected to meet the following basic requirements.

(1) Its size should be sufficient to accommodate the workpieces and heating devices.

(2) Its volume should be kept to a reasonable limit so as to minimize the pump-down time.

(3) The welding zone should be easy to reach.

(4) The chamber should be fitted with devices capable of sealing it in the least possible time.

(5) All components should be mechanically and thermally strong, and retain heir shape and rigidity under service conditions.

(6) The chamber and the associated vacuum system must be vacuum tight.

(7) As a corollary of the requirement in (6) above, the chamber must be fabricated from gas-tight materials, all welded and brazed joints must be vacuum-tight, its demountable vacuum joints should be properly designed, and its internal surfaces must be carefully machined and otherwise treated.

(8) The chamber should suit the expected physical conditions stemming from the use of particular heating sources (for example, a high electric potential, an r.f. magnetic field, etc.).

From the above list it is obvious that it would be next to impossible to develop a design that would suit all the requirements at a time. In each particular case, a compromise is sought, depending on the field of application, the size and constructional features of the workpieces, and the scale of operation.

More specifically, a diffusion-bonding vacuum chamber (Fig. 4-1) consists of a housing *1*, a cooling jacket *2*, a charging port *3*, an observation port *4*, an intermediate spindle *5* fitted with sealing elements, a heating device *6*, and electric lead-ins *7*. The number of some of the elements may vary from chamber to chamber.

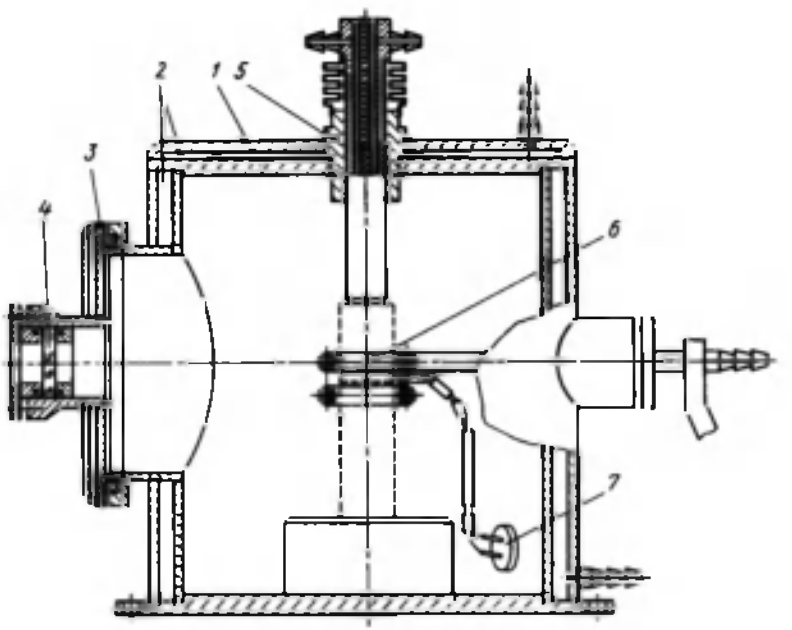


FIG. 4-1 Vacuum chamber

In addition to the basic components essential to normal operation, chambers may be fitted with optionals, such as lights inside the chamber, extra cooling devices, workpiece mounts, etc. [11].

BASIC TYPES OF BONDING CHAMBER. Arbitrarily, the vacuum chambers used in diffusion-bonding units may be classed into two major groups, namely general-purpose and special-purpose. By this classification, a general-purpose chamber is that which can accommodate and handle a large variety of workpieces provided their overall dimensions are within the chamber size. A special-purpose chamber is that which is tailored to cater for a particular kind of job. In this book they will be taken up in connection with special-purpose diffusion-bonding units.

General-purpose diffusion-bonding chambers may be divided into cylindrical, rectangular, and skeleton.

Cylindrical chambers. Most widely used in vacuum work, cylindrical chambers are often employed in diffusion bonding. They retain their shape and size under uniform loads and lend themselves readily to streamlined fabrication as they are made of seamless drawn or rolled tubes. Cylindrical chambers may use any one of many types of ports (and lids) through which workpieces are charged into and withdrawn from a chamber (Fig. 4-2). In fact, the design of ports and covers will usually affect that of the chamber and of the diffusion-bonding unit as a whole. Ac-

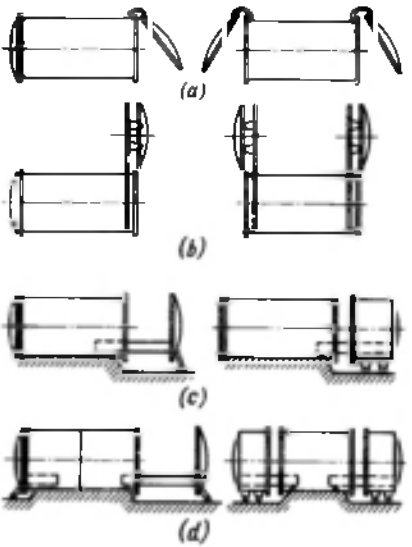


FIG. 4-2 Principal types of cylindrical vacuum chamber

cordingly, cylindrical chambers may be classed into those with hinged lids (Fig. 4-2*a*) designed to accommodate small and medium-size parts; those with swung-out lids (Fig. 4-2*b*) capable of accommodating large-size weldments; and those with lids rolled out along the chamber axis (Fig. 4-2*c* and *d*). In the latter case, the lid carriages are positively coupled to the chamber carriage. A distinction of this latter type is that one may use a variety of hoisting and conveying facilities which is especially important in handling bulky products.

Rectangular chambers. This type is advantageous in that the chamber can readily be expanded by adding extra sections in the desired direction. In this way, the chamber volume can be better utilized, its components replaced at will, and the chambers themselves unified and standardized. As compared with the cylindrical type, rectangular chambers take more labour to fabricate them. Also, the chamber walls have to be made sturdier and fitted with stiffeners in view of the likely deformation.

Skeleton chambers. A chamber of this type (Fig. 4-3) is built around a skeleton one side of which is a solid plate or slab. With it, the chamber is attached to the frame of a welding unit and connected to a pumping system. The other sides of the skeleton receive hermetically sealed demountable or swung-out lids whose shape and design depend on the intended application of the associated welding unit. The types of lids for skeleton chambers are many and diverse. Light-weight lids are preferably of demountable construction (Fig. 4-3*a*). Small and medium chambers are fitted with hinged lids capable of rotation about a horizontal axis (Fig. 4-3*b*) or a vertical axis (Fig. 4-3*c*). This design is attractive because the chamber can readily be expanded by attachment of extra sections in the desired direction. Examples of expanded skeleton chambers are shown in Fig. 4-3*d*. A chamber with an only frontal

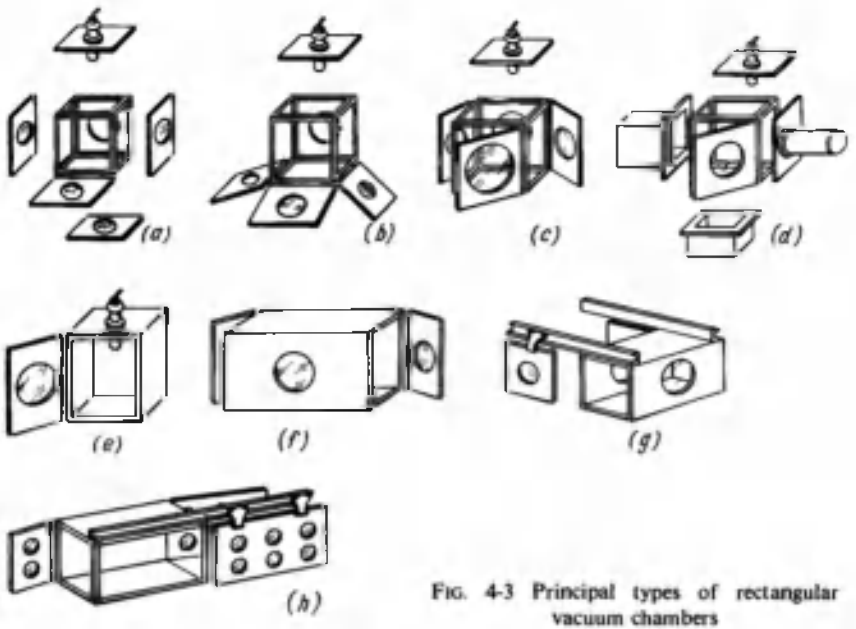


FIG. 4-3 Principal types of rectangular vacuum chambers

lid (Fig. 4-3*e*) has an all-welded sheet-steel shell. Such a chamber can be moved about with difficulty, but is extremely simple to fabricate and has a minimal number of demountable vacuum joints. A chamber with two side lids (Fig. 4-3*f* and *g*) is a horizontally extended rectangular all-welded shell. With this design, the chamber can only be expanded along its length. The lids may be hinged (Fig. 4-3*f*) or mounted on roller-supported carriages which move on horizontal guides built into the chamber shell (Fig. 4-3*g*). A chamber with two hinged lids and one roll-out lid (Fig. 4-3*h*) is a happy combination of a chamber with hinged lids and a chamber with a roll-out lid.

With the chamber designs examined above, one has to reserve a sizeable floor area so that the lids giving access to the chamber insides can be conveniently opened. Also, the swung-out lids pose additional difficulties in plant layout and operation. This limitation is avoided in the chamber design shown in Fig. 4-4. It consists of a stationary shell in which all the necessary ports are made, and a rotating inner sleeve set up on rollers. The space between the shell and the sleeve is hermetically sealed by elastic tubular gaskets inflated with compressed air. In this design, the rotating sleeve which acts as a lid takes up no extra floor area in the shop. The chamber can be set up horizontally or vertically.

MATERIALS FOR VACUUM CHAMBERS. The materials that go to make chambers for industrial diffusion-bonding units should meet a number of requirements which may be stated as follows.

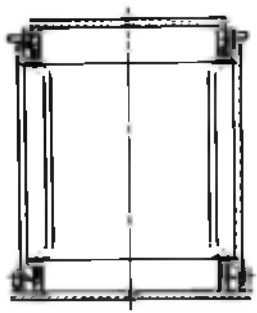


FIG. 4-4 Cylindrical vacuum chamber with a rotating sleeve

(1) A maximum vacuum tightness even at small thicknesses.
(2) A minimum outgassing even at elevated temperatures.
(3) Good machineability and weldability by various electric welding processes.
(4) Corrosion resistance.

In the Soviet Union, shells for diffusion-bonding units are most commonly made of structural carbon steels, such as steel-20, and stainless steels, such as 12Х18Н10Т. Inexpensive and more readily machineable, carbon steels are predominantly used to fabricate medium and large chambers. As many years' experience has shown, they have good resistance to corrosion as well, because the vapours of vacuum fluids condense on the chamber wall in operation and form a thin oil film. This film has a negligible effect on the pump-down time to a pressure of 1.3×10^{-2}-1.3×10^{-3} Pa, but successfully protects the chamber surfaces against corrosion.

Chambers for microjoining and precision work where residual magnetic fields might impair the quality of products, and also chambers that are washed after bonding by corrosive media are fabricated from stainless steels, such as 12Х18Н10Т. As an alternative, use may be made of a two-layer material, such as a carbon steel clad on the inside with a thin layer of stainless steel. This saves the expensive stainless steel and ensures a high-quality inner surface in the chamber.

The actual fabrication of diffusion-bonding chambers, notably preparation of blanks, their machining, joining, etc., differs very little from that adopted in general vacuum engineering. Special emphasis is placed, however, on the proper assembly and joining of shell blanks, and the finishing of a complete shell. The objectives of the finishing operations are to relieve locked-up welding stresses as they might cause deformation after machining, to restore the planeness of the sealing surfaces, and to improve the finish of all inner surfaces. Ordinarily, locked-up welding stresses are relieved by annealing or ageing (natural or artificial). Following that, the sealing surfaces and, if necessary, all internal surfaces are polished electromechanically or chemically. The mating surfaces of flanges must be finished to a roughness of not over 1.25 μm, and all internal surfaces to a roughness of at least 20 μm.

4.4 Vacuum Pumping Systems

In diffusion-bonding units, vacuum pumping systems serve to build up and maintain the desired vacuum in the bonding chambers.

A typical vacuum pumping system consists of pumping facilities, valves, vacuum pipework, and instrumentation. As recommended by the Fundamental Research Laboratory for Vacuum Diffusion Bonding, all diffusion-bonding units in the USSR are grouped into three classes which differ by the extent to which the materials are shielded against exposure to the atmospheric gases. Their layouts are given in Table 4-11.

For industrial diffusion bonding in high vacuum, use is most often made of layout I (see Table 4-11). It consists of a high-vacuum pump assembly *1*, vacuum valves *2*, a solenoid-operated safety valve *3* which cuts off the system in the case of a sudden power failure, air valves *4*, a fore-vacuum pump *5*, a trap *6*, vacuum-gauge sensors *7*, and vacuum-gauge indicators.

For larger chambers (over 20 m^3 in volume), preference is given to layout II. As compared with layout I, it includes a booster pump *8* with a maximum intrinsic speed in the pressure range 13.3-0.13 Pa. Sometimes, a second fore-vacuum pump may be installed.

In cases where a heavy outgassing is expected, layout III is preferable. Here, a booster pump, *8*, is connected in series with the main pump assembly. With this arrangement, larger chambers can be exhausted and a lower pressure obtained in the vacuum system.

The pumping systems using diffusion pumps (layouts I, II, and III) suffer from a major limitation: in the course of pumpdown the vacuum fluids used contaminate the chamber space. In cases where diffusion-bonded weldments are to operate in corrosive media, the joints in them must show the utmost in corrosion resistance, and this calls for the use of oil-free vacuum. As a way out, it is usual to prevent the creep of vacuum fluid vapours by provision of cryogenic or sorption traps, *6*. This, however, solves the problem only in part. Therefore, the usual practice in the most critical applications is to employ oil-free pumping facilities (layout IV) where the main pumps are sputter-ion (cold-cathode getter-ion) pumps, arc-discharge pumps, plain getter pumps, or turbomolecular pumps (at *10* in the figure) which do not use any vacuum fluids. Low-throughput diffusion pumps are then used sometimes to pump out inert gases.

For studies into diffusion bonding in an ultra-high vacuum, use is made of layout V. Here, all joints are metal-gasketed, and the system is baked out to 723 K in order to degas all internal surfaces. This increases the required pump-down time.

Diffusion-bonding units of the rotary-table and conveyor-belt types use several independent pumping systems each of which serves a chamber of its own (layout VI).

VACUUM PUMPS. Of the many known types of vacuum pumps commercially available in the Soviet Union, diffusion-bonding units are equipped with mechanical (forevacuum) pumps and pump assemblies, vapour-jet pumps and pump assemblies, and sorption pumps.

TABLE 4.11

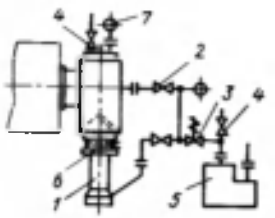
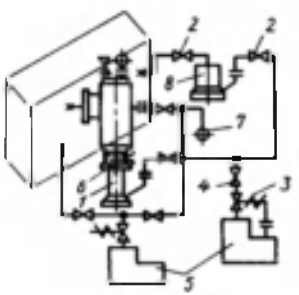
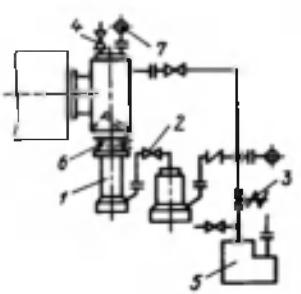
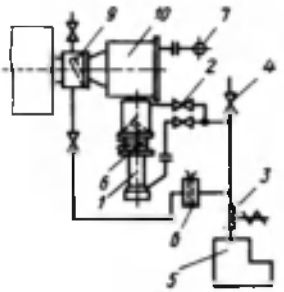
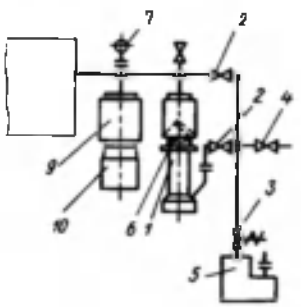
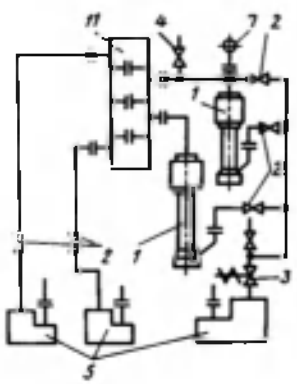
Vacuum pumping system	Vacuum
I 	1.3×10^{-4} Pa (rubber gasketing)
II 	Same
III 	1.3×10^{-4} Pa (rubber gasketing) 1.3×10^{-5} Pa (PTFE or metal gasketing)

TABLE 4-11 (*concluded*)

Vacuum pumping system	Vacuum
IV 	1.3×10^{-4} Pa (rubber gasketing) 1.3×10^{-6} Pa (metal gasketing)
V 	6.5×10^{-7} Pa (metal gasketing)
VI 	1.3×10^{-2}-1.3×10^{-3} Pa

The mechanical group includes type VN oil-sealed pumps, type RVN Roots blower pumps, and turbomolecular pumps. They serve to rough-pump the associated chamber (to a pressure of 1 011 hPa-1.3 Pa), and to back up high-vacuum pumps. Their performance data are presented in Table 4-12.

Type RVN pumps are used in diffusion-bonding units in cases where outgassing is heavy. They have a high intrinsic speed in the pressure range $1.3\text{-}1.3 \times 10^{-1}$ Pa and stand midway between type VN pumps and high-vacuum diffusion pumps. Turbomolecular pumps are designed for operation at high and ultra-high vacuum ($1.3 \times 10^{-2}\text{-}1.3 \times 10^{-8}$ Pa). They are used seldom, such as when the conditions prevailing in outer space are simulated.

By far the most widely used pumps in Soviet-made diffusion-bonding units are type BN vapour booster pumps, type N high-vacuum oil-vapour pumps, and type VA pump assemblies. The vapour booster pumps are convenient in pumping out large quantities of gas at a high rate in the pressure range 1.3-0.13 Pa. High-vacuum pumps and pump assemblies (Table 4-13) are most reliable in the pressure range $1.3 \times 10^{-4}\text{-}6.65 \times 10^{-4}$ Pa. An advantage of these pumps is the fact that their intrinsic speed remains practically unchanged over a wide range of operating pressures.

Sorption pumps are used to produce high and ultra-high vacua free from oil vapour. Since they use no pump fluid, the evacuated space is free from contamination by pump fluid vapours. A disadvantage of these pumps is a low throughput in pumping out large volumes with a high content of organic compounds (oil, etc.), and also in pumping out inert gases.

Zeolite and sputter-ion (cold-cathode getter-ion) pumps and assemblies have not found an appreciable application in diffusion bonding because they have a low speed.

VALVES. These are extremely important components of any vacuum system. In terms of requirements they should meet, vacuum valves differ a good deal from their counterparts used in hydraulic and pneumatic systems. Among other things, they should be perfectly vacuum-tight (especially when they are used in high and ultra-high vacuum systems), show a maximum throughput when open (that is, offer a minimum resistance to the flow of the pumped gases), permit disassembly and repair, use no greases or gaskets from materials with a high vapour pressure, be free from hard-of-access and difficult-to-pump pockets, and be fabricated from materials which do not corrode and adsorb gases only slightly. Valves for ultra-high vacuum systems must permit repeated baking to 573-873 K for degassing.

In the Soviet Union, the vacuum pumping systems of diffusion-bonding units are at present using solely metal valves with gaskets fabricated from vacuum rubber, PTFE, Viton, copper, and some other materials. Bakeable metal-gasketed valves are used on a limited scale, mainly in research work. Safety valves are mostly of the solenoid-operated elbow type. All of the valves can be installed in any position and will provide a tight seal at atmospheric pressure on any side of the valve plate or disk. In more detail, vacuum valves are described in the literature on the subject.

TRAPS. Sometimes vacuum pumping systems are fitted with traps in order to improve the ultimate pressure in the chamber and to prevent the contamination of the

TABLE 4-12

Particulars	VN-2MG	VN-1MG	(VN-4G) VN-7G, VN-7M	VN-6G (VN-6MG)	VN-300 (VN-300M)	VN-500 (VN-500M)	VN-10-2
Number of stages	2	2	1	1	1	1	2
Speed in pressure range 1 011 hPa 1.33 Pa, $l \cdot s^{-1}$	5.8	16.5	45	120	300	500	6
Ultimate pressure, Pa:							
total, with gas ballasting	33.3	7.3	133	133-200	133	133	4
same, without gas ballasting	3.06	3.06	4	6.65	6.65	6.65	6.65
partial, in terms of air	4×10^{-1}	4×10^{-1}	6.65×10^{-1}	1.33	1.33	1.33	6.65×10^{-3}
Rotor speed, rpm	525	500	500	360	260	210	540
Flow rate of cooling water, l/h	Air-cooled		2300	3000	5500	6500	
Qnty of oil poured in pump, l	2	3.8	15	55	80	85	0.8
Power rating of 3-phase induction drive motor, kW	1.7	2.8	7.0	20.0	40.0	55.0	1.0
Overall dimensions of pump, mm:							
length	822	954	1370(1490)	1560(1500)	2975	2910	752
width	486	580	770	970(980)	1510(1705)	1850	342
height	575	745	1300	1790	1800	1535	680
Pump weight, kg	187	290	690(622)	1557(1500)	4226(2613)	1535(4078)	120

Notes. 1. The pump fluid is VM-4 or VM-6 vacuum oil.
2. The figures in the parentheses refer to modernized pumps.

TABLE 4-12 (*continued*)

Particulars	VN-10-1	VN-20-2	VN-20-1	VN-40-2	VN-40-1	VN-75-1	VN-150-1
Number of stages	1	2	1	2	1	1	1
Speed in pressure range							
1 011 hPa-1.33 Pa, $l\,s^{-1}$	9	12	18	25	40	75	150
Ultimate pressure, Pa:							
total, with gas ballasting	106	4	106	1.33	106	106	106
same without gas ballasting	6.65	6.65×10^{-1}	5.65	6.65×10^{-1}	6.65	6.65	6.65
partial, in terms of air	6.65×10^{-1}	2.66×10^{-3}	6.65×10^{-1}	2.66×10^{-1}	6.65×10^{-3}	2.66×10^{-1}	2.66×10^{-1}
Rotor speed, rpm			540			475	450
Flow rate of cooling water, l/h		Air-cooled					
Qnty of oil poured in pump, l						600	900
Power rating of 3-phase induction drive motor, kW	1.4	1.5	2.5	2.3	3.7	14	25
Overall dimensions of pump, mm:	1.1	1.5	2.2	4.0	5.5	9	13
length	752	752	752	872	872	1430	1430
width	360	415	597	597	597	650	1183
height	668	680	668	690	678	1165	1175
Pump weight, kg	110	651	170	290	285	770	940

TABLE 4-13

Particulars	VA-01-1	VA-05-1	VA-2-3	VA-5-4	VA-8-4
Average pump speed, $l\ s^{-1}$, for air in pressure range 1.3×10^{-3}-2.5×10^{-2} Pa	25/50	100/250	500/1000	1200/2200	2500/4000
Max. outlet pressure, Pa	9.75/9.75	9.75/13.3		13.3/13.3	
Heater rating, kW	0.5/0.5	1.0/1.0	1.2/1.2	2.0/2.0	2.8/2.8
Qnty of oil poured in, l	0.05/0.05	0.5/0.5	1.0/1.0	1.5/1.5	4.0/4.0
Capacity of nitrogen trap, l	0.5/-	0.5/-	1.5/-	1.5/-	1.5/-
Height to axis of inlet port, mm	630/600	870/820	1120/1045	1310/1230	1535/1450
Height of assembly, mm	760/700	1080/1030	1320/1250	1650/1570	1990/1900
Weight, kg	47/43	90/85	200/190	330/315	500/476
Flow rate of cooling water, $l\ h^{-1}$	50	120	200	400	500
Type of high-vacuum pump	N-1S	N-5S	N-2T	N-5T	N-8T
Type of roughing pump (recommended)	VN-461M	VN-2	VN-2	VN-1	VN-1

Note. The figures in the numerator apply to a pump with a nitrogen trap ($p = 6.65 \times 10^{-5}$ Pa), and the figures in the denominator to a pump without a nitrogen trap ($p = 6.65 \times 10^{-4}$ Pa).

vacuum space with vapours of pump fluids and cracking products. Most frequently, these are cryogenic and sorption traps. The former depend for their operation on the condensation of pump fluid vapours on a cryogenic surface. The latter utilize the absorption or adsorption of pump fluid vapours on an activated material. Among cryogenic traps the most commonly used type is the nitrogen cooled trap. In Soviet practice, nitrogen cooled traps are usually fitted in vacuum systems incorporating high-vacuum pump assemblies.

Figure 4-5 shows the mass spectrum of residual gases in the chamber of a typical diffusion bonding unit, fitted with a nitrogen cooled trap. The chamber made of killed low-carbon steel is 0.25 m^3 in volume and has rubber gaskets. Its pumping system consists of a type VN-1 fore-vacuum pump and a type VA-2-3 high-vacuum pump assembly. The residual pressure in the chamber is mainly due to high-molecular-weight hydrocarbons. Installed at the exit from the oil-vapour pump, the trap more than halves the partial pressure of the high-molecular-weight hydrocarbons and water. The partial pressure of oxygen is reduced only slightly (Fig. 4-5*b*), but it can be brought down effectively by titanium sorption traps or titanium sorption pumps. With them (Fig. 4-6) no oxygen peak is observed. In the operation of titanium-based devices it is characteristic that the partial pressure of hydrogen sharply rises when the titanium evaporator is operating (Fig. 4-6*b*), but within 20 to 25 s after the evaporation is stopped, no hydrogen peak is observed.

INSTRUMENTATION. This primarily refers to vacuum gauges. They may be classed into absolute instruments and inferential instruments. The former include positive-displacement, elastic-element, liquid-column and McLeod gauges. They

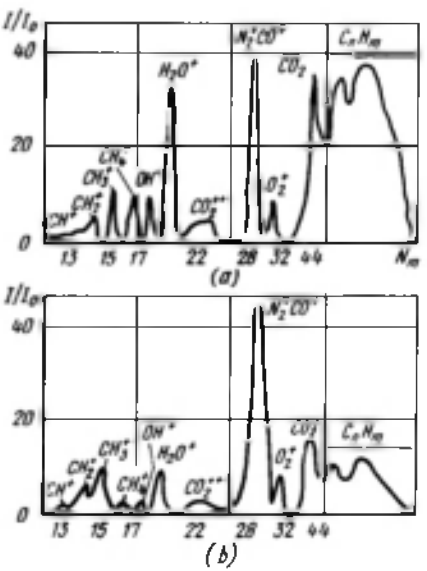


FIG. 4-5 Mass spectra of residual gases after exhaustion by (*a*) oil-vapour pump; (*b*) oil-vapour pump fitted with a nitrogen trap

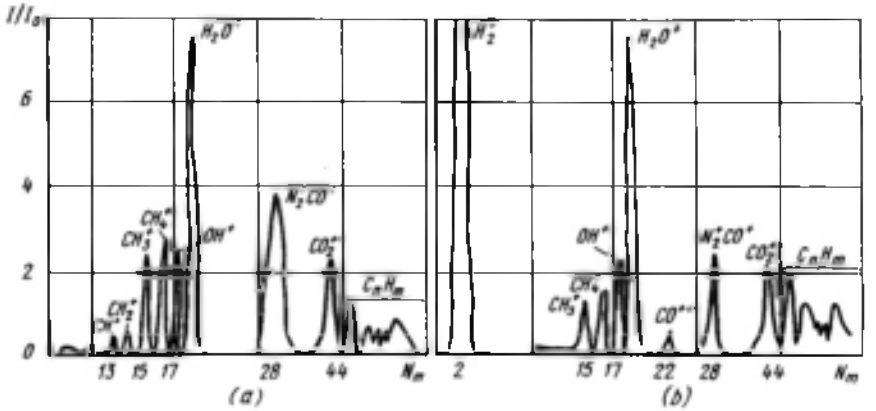


FIG. 4-6 Mass spectra of residual gases after exhaustion by a titanium pump and an oil-vapour pump of low throughput: (*a*) evaporator OFF; (*b*) evaporator ON

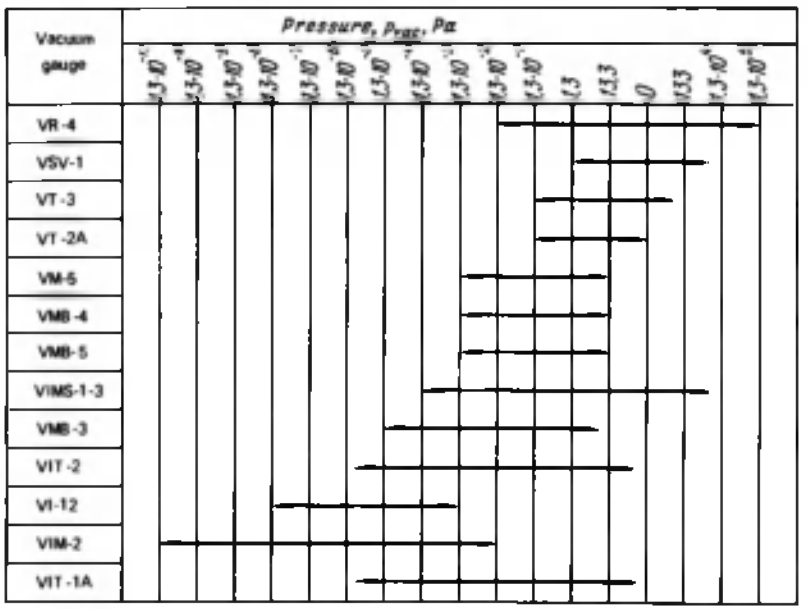
have not found any appreciable use in diffusion-bonding equipment and will not be taken up in this book. Gauges in the inferential group utilize in their operation the pressure dependence of some physical processes taking place in a vacuum. Most frequently, diffusion bonding units are fitted with thermal-conductivity, hot-cathode and cold-cathode ionization gauges. As their name implies, thermal-conductivity vacuum gauges depend for their operation on variations in the thermal conductivity of a gas with pressure. In hot-cathode ionization gauges, use is made of the ionization of the residual gas by a stream of electrons or alpha-particles. In cold-cathode ionization (or sputter-ion) vacuum gauges, a measure of pressure is provided by the current of the discharge that occurs in a vacuum in the presence of an electric and a magnetic field. A summary on the ranges of some of the Soviet-made vacuum gauges is given in Table 4-14.

Some vacuum gauges are fitted with interlocks which generate a signal according to the pressure reached, others are ganged up with potentiometers which record pressure signals on a strip chart. Those fitted with interlocks are primarily used for safety purposes (for example, they will turn on a heating device only when the chamber has been pumped out to a pressure of 1.3×10^{-2} Pa). Those ganged up with a potentiometric recorder are incorporated in automatic pumping systems. Where appreciable currents have to be switched, the interlock relay(s) of a vacuum gauge may be used to trip more powerful relays in a control system.

It should be remembered that the accuracy of inferential gauges, especially those fitted with interlocks, is rather low, being anywhere between 10% and 60% of the indication.

Testing of components for vacuum tightness is based on leak hunting with the aid of mass spectrometers. Most often, resort is made to helium leak detectors which can readily detect minute amounts of helium finding its way through discon-

TABLE 4-14



tinuities in welded joints. In the arrangement shown in Fig. 4-7, helium is blown over an evacuated component and the detector is placed inside the latter (this is known as vacuum testing).

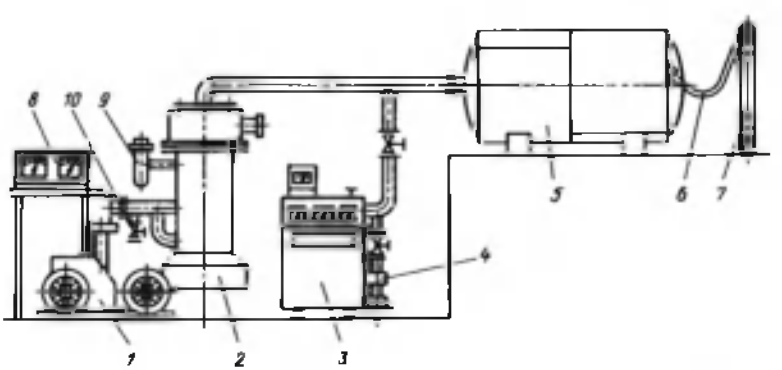


FIG. 4-7 Set-up for helium leak hunting by vacuum testing:
1 — mechanical pump; *2* — oil-vapour pump; *3* — leak detector; *4* — helium standard leak; *5* — vessel under test; *6* — vacuum rubber hose; *7* — compressed helium cylinder; *8* — vacuum gauge; *9* — nitrogen trap and vacuum-gauge sensors; *10* — shut-off valve

4.5 Basic Types of Heat Sources and Methods of Heating

The various methods of heating the workpieces in diffusion bonding in vacuum may be classed into two broad groups. One includes those which utilize external heat sources, with heat transferred to the workpieces by radiation or thermal conduction. The other includes those in which heat is generated in the workpieces themselves by conversion of electricity into thermal energy [2, 4, 5, 7].

RADIATION HEATING. In this case, a heat source may be placed inside or outside the vacuum chamber. The maximum permissible temperature for radiation heating depends on the thermal stability of the chamber material. In sketch form, several arrangements using radiation heating are shown in Fig. 4-8. The work, *1*, is set up on a mount, *2*, inside the vacuum chamber, *3*, and is heated by radiation from a heater, *4*, placed outside (Fig. 4-8*a*) or inside (Fig. 4-8*b*) the vacuum chamber.

When the chamber is fabricated from fused quartz or a similar material, the maximum permissible temperature for the heating of workpieces is limited by the softening point of the material and the formation of porosity which might disturb the vacuum. The usual rate of temperature rise is 40 to 50 K min^{-1}. When diffusion bonding is done in a quartz tube 25 mm in diameter and with a wall thickness of 2.5 mm, molybdenum disilicide heaters can raise the temperature to 1 473 K in a matter of 20 to 30 min. The rate of temperature rise can be controlled by varying the voltage applied to the heater. In practice, the heaters, *4*, are usually placed inside the chamber, in which case the workpieces can be heated to 2773-3273 K. This temperature can be obtained with tungsten or graphite heaters. In the temperature range 1633-2633 K it will suffice to use heaters made of molybdenum or titanium. Where the heating temperature is 1 273 K, the purpose can well be served by Ni-Cr alloy heaters.

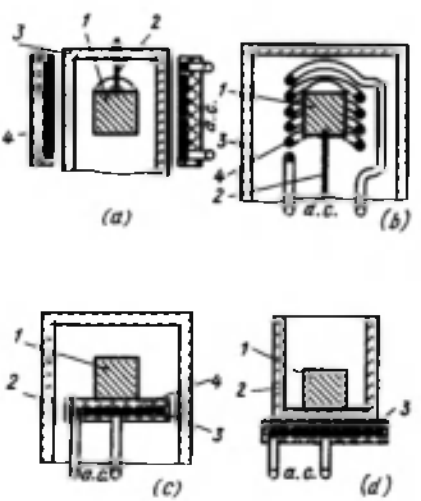


FIG. 4-8 Heating of parts in vacuum by radiation and heat conduction

In using the above types of heater, it should be remembered that the heater material might vaporize and deposit on the workpieces, a feature undesirable in many instances. This is avoided in the arrangement shown in Fig. 4-8*c* where the specimen, *1*, is heated by a heater, *3*, placed inside the chamber, *2*. So that the workpiece could not melt and be welded to the heater if they were in direct contact, the latter is given a thin coat of aluminium oxide, *4*, which separates them.

In the arrangement shown in Fig. 4-8*d*, the workpiece, *1*, is placed inside the chamber, *2*, and heat is supplied by an electric furnace, *3*, placed outside. Now heat is mostly supplied by thermal conduction, although radiation from the surface of the hot chamber also plays an important part.

The amount of heat, q (in W cm^{-2}), transferred to a specimen by radiation from the surface of a heater obeys the law experimentally induced by Stefan and subsequently derived theoretically by Boltzmann

$$q = 5.72K_r[(T_h/1000)^4 - (T_s/1000)^4]$$

where K_r is the radiation constant which depends on the physical properties of the material and its surface condition. For a black body $K_r = 1$, for a polished molybdenum heater K_r is approximately 0.2, for steel with an oxidized surface it is about 0.8, and for steel with a polished surface, about 0.3. The difference in temperature between the heater (T_h) and the specimen (T_s) increases with increasing rate of temperature rise and depends on the condition of heat transport. The difference also increases with increasing separation between the heater and the specimen and also when a barrier is placed between them. The rate of temperature rise when an external heat supply is used (given the same power rating) depends on the size, thermal conductivity and specific heat of the heater (radiator) and specimen. The rate of temperature rise goes down as the size and, in consequence, the mass of the specimen are increased.

Exact calculations for the above arrangements are difficult to make because one has to account for the effect of radiation, shields and similar elements incorporated in the chamber design. This is why the above general considerations must be supplemented by experiments.

A major distinction of radiation heating from an external source is that the workpieces may be of any material — conductors (metals and alloys), semiconductors, and dielectrics.

RESISTANCE HEATING. Now the required heat is supplied by the passage of an electric current through the workpieces themselves. The pieces are in direct contact with the current-source. The rate of heating depends on the resistance of the specimen, R_s, and the rms value of the current, I_{rms}, passing through the specimen. The amount of heat, Q (in cal), generated by the passage of current can be found by Joule's law:

$$Q = 0.239I_{rms}^2R_st$$

where t is the time of current passage in s.

In resistance heating the maximum temperature of the work is only limited by its melting point. An essential requirement in resistance heating is the provision of a reliable physical contact between the work and the electrodes conveying the current.

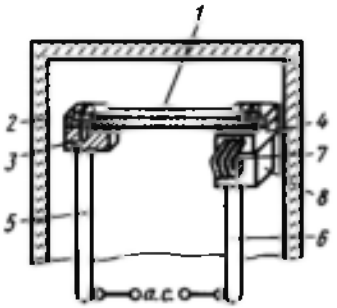


FIG. 4-9 Resistance heating of parts in vacuum

An arrangement for resistance heating is shown in Fig. 4-9. The workpiece, *1*, placed inside a vacuum chamber, *2*, is clamped in terminals, *3* and *4*. Terminal *3* is made fast to an electrode, *5*, whereas terminal *4* is connected to a second electrode, *6*, by a copper pig-tail, *7*, and a copper block, *8*. Provision of a flexible pig-tail is important as it avoids straining the work due to the volumetric changes occurring in the course of heating and cooling. The two electrodes are usually hollow tubes cooled with running water. They enter the vacuum chamber through well-gasketed seals. In this arrangement, the best type of current source is the transformer of a resistance welding machine[1] which also includes a pair of electrodes connected to clamps operated by hand or automatically. When copper electrodes are used, heat is generated owing to the high contact resistance at the junction of the electrodes and the work. Given the same current, the contact resistance depends on the pressure applied, that is, the area and closeness of electric contact. After a weld is complete, the contact resistance sharply decreases, and further heating occurs due to the resistance of the work, which increases with rising temperature.

Apart from copper, the electrodes can be made of graphite and tungsten, in which case the materials to be heated may widely differ in thermal conductivity and resistance. A further advantage is that heat can be supplied to hard-to-reach spots. Which type of electrode suits a given material best can be established by experiment, using various combinations and various geometry. With graphite or carbon electrodes which are relatively fragile, a lower pressing load should be used than in the case of electrodes made of high-temperature alloys and steel.

INDUCTION HEATING. In this case, the workpiece to be heated is placed in the high-frequency electromagnetic field set up around an inductor by a source of high-frequency current. A distinction of induction heating is that electric energy is transferred from inductor to work over a distance of a few centimetres, without any actual contact between them. Heat is generated within the work by the circulating eddy currents induced by the applied magnetic field. The current thus induced is

$$I = E/R = 4.44fw\Phi \times 10^{-8}/R$$

[1] This may be a spot, seam or projection resistance welding machine.

where R is the total apparent resistance of the workpiece in ohms, E is the emf in volts, f is the frequency of the applied electric current in hertz, w is the number of turns in the inductor, and Φ is the magnetic flux threading the workpiece in webers. As is seen, the induced current is directly proportional to the frequency of the applied current, the number of turns in the inductor, and the magnetic flux, and inversely proportional to the resistance of the workpiece. This current is in the direction opposite to the flow of current in the inductor.

The density of the induced current in the workpiece, as in any conductor carrying an alternating current, is not the same over its cross section due to the influence of three effects: the skin effect, the proximity effect, and the coil effect. The skin effect consists in that the density of the alternating current in the conductor is a maximum at its surface (skin) and sharply decreases on moving towards its centre. The skin effect becomes ever more pronounced with rising frequency of the applied current and increasing electric conductivity and permeability of the conductor material. The proximity effect consists in that the distribution of the current induced in two closely spaced conductors depends on the direction of current flow in those conductors. When the current flow is in the same direction in both conductors, the induced current is a maximum on the opposite sides. When the current flow in one conductor is opposite to that in the other conductor, the current density is a maximum on the adjacent sides. In the former case, the magnetic lines of force of the two fields between the neighbouring conductors act in opposite directions and weaken each other. In the latter case, the magnetic lines of force between them are in the same direction, and this serves to build up the magnetic flux density between the neighbouring conductors. This effect gains in magnitude as the spacing between the conductors is decreased. The coil effect occurs due to the dissymmetry of the electromagnetic field around a conductor when it is made into a coil: the lines of force are spaced closer together (crowded) inside and farther apart outside the coil. The three effects become more pronounced with increasing frequency of the applied alternating current.

Acting together, the three effects may augment or diminish each other. For example, when a cylindrical tube is heated on the outside, the coil effect is augmented by the proximity effect between the inductor and the tube. Because the current induced in the tube is opposite to that flowing in the inductor, the current density and, in consequence, the amount of heat generated will be a maximum on the outer surface of the tube. If we place an inductor inside a cylindrical tube, the coil effect will moderate the skin and proximity effects, and the amount of heat thus generated will be smaller.

As already noted, the induced current depends also on the resistance and permeability of the work material, and these are in turn dependent on the temperature and duration of heating. This is why the manner in which a part is heated will vary as its temperature goes up. In the case of steel parts, the rise in temperature leads to an increase in the resistance, whereas the permeability changes but little, and sharply falls only near a temperature of 1 073 K (the Curie point). This is why, in heating steel parts, the skin effect is strongly pronounced until the surface layer reaches the critical temperature. In that layer, the skin effect is less

noticeable because the electric conductivity is now low and the permeability is very low. As the layer widens, a progressively greater proportion of heat passes into the bulk of the component, until the resistance of the steel goes so high that all of the field energy is converted to heat. With paramagnetic metals (copper, aluminium, brass, bronze, etc.) the rise in electric resistance leads to a more uniform distribution of the induced current and, as a consequence, of heat. The induction heating of metals may use frequencies from 50 to 2×10^6 Hz. For substantial components, it is common to operate at a frequency of 8 000 Hz supplied by machine generators. At that frequency, heating proceeds more slowly and more uniformly owing to higher thermal conductivity and the reduced difference in temperature between the surface and the bulk of the component.

The alternating electromagnetic field is set up around the component by the high-frequency alternating current flowing around the inductor. The shape and size of the inductor are chosen to suit those of the workpieces. As a rule, inductors are fabricated from circular or rectangular copper tubes cooled by running water in operation. Sometimes, an inductor may be given a more complex form by combining it from tubes, bars and sheets. Inductors may be made one-piece or splittable, single- or multiturn, externally or internally applied to the work. The equations involved in inductor design are mostly approximate, so it is customary to rely more on past experience and to refine the design in subsequent usage. The magnitude of induced current depends on the size of the inductor and on the spacing between the inductor and the component. The spacing, or gap, usually varies according to the shape of the workpiece and the desired rate of heating: with a wider gap the rate of heating slows down. Small gaps are convenient for thin-walled pieces. Wider gaps, up to 20 mm, are used in cases where it is desired to join parts differing in thickness or having irregular shapes, so as to avoid the likely melting of edges and of high spots. Unfortunately, wide gaps impair the efficiency of the inductor. On the other hand, gaps smaller than 2 mm might lead to short-circuits between the inductor and the workpiece. To avoid this, it is usual to insulate the inductor turns with enamel or asbestos cord impregnated with soluble glass. In multiturn inductors the turns are usually arranged in series, so the same current is induced in all the turns. Where one of the turns is to be used for heating a part of the component, it may be connected in parallel. With parallel connection, the currents in the turns may be different.

Multiturn inductors are advantageous in that they better utilize the generator capacity, but they produce a banded pattern due to nonuniform heating which may finally result in cracking. One way to avoid banding is to make multiturn inductors from rectangular copper tubes wound with the turns nearly touching. The banding effect may be mitigated by increasing the gap between the inductor and the work. In flat workpieces, the proximity effect causes the induced current to trace out a path following the shape of the inductor.

The depth of the surface layer within which some 90% of the total heat is produced by electric current is known as the penetration. As studies have shown, it is given by

$$h = 5.03 \times 10^4 \sqrt{\rho/\mu f}$$

where ρ is the resistivity of the material at the specified temperature in Ω cm, μ is the permeability of the material, and f is the current frequency, Hz.

When, say, a specimen of low-alloy constructional steel is heated in the range 293-1273 K, its ρ and μ change, and the penetration increases by a factor of 25 or 30. Characteristically, the penetration is an inverse function of the square root of the frequency, and the work diameter to penetration ratio ranges anywhere between 4 and 10. In the circumstances, the work can be heated at a high rate, with a high efficiency, and a high energy concentration. The generator capacity is chosen to suit the size of the work to be handled.

ELECTRON-BEAM HEATING. A vital pre-requisite for electron-beam heating is a sufficiently high vacuum in the process chamber. Otherwise, the electrons of the beam, on travelling from the source to the target, would collide with the remaining gas molecules, ionize them, lose some of their energy, and scatter. The mean free path should be at least equal to the distance from the electron source to the workpiece. Ordinarily, the mean free path is given by

$$\lambda = kT/\pi\rho^2 p$$

where $k = 1.38 \times 10^{-23}$ J K^{-1} is Boltzmann's constant, ρ is the theoretical radius of a gas molecule (for air, $\rho = 1.84 \times 10^{-8}$ cm), and p is the pressure of the gas, Pa.

A plot of the mean free path as a function of air pressure in a chamber at 293 K is shown in Fig. 4-10. As is seen, the vacuum in an ordinary chamber should be maintained at not below 6.65 Pa.

The electron source is made as simple as practicable. Basically, this is a combination of a cathode producing a cloud of free electrons and electrodes which set up an electric field in the cathode-anode space. The electric field shapes the electrons into a beam which is accelerated towards the target. If the workpiece is made the anode, there is no need for any auxiliary electrodes to focus the beam. When the anode is an aperture plate, or slit, which allows electrons to enter the process chamber, it is usually followed by an electromagnetic system to focus the electron beam and, if necessary, to deflect it. Systems with the anode made separately from the workpiece and in the form of an aperture plate are more common than those in which the workpiece is the anode.

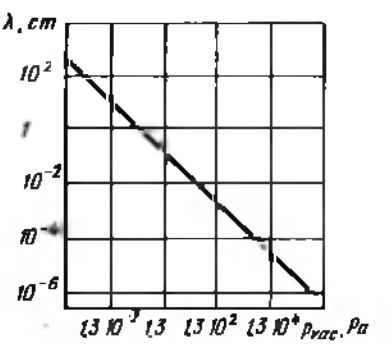


FIG 4-10 Mean free path of electrons in air as a function of pressure in the chamber

In electron-beam heating units, it is usual to employ thermionic cathodes. Experiments have been made to use the plasma of a gas discharge as the cathode, but the current in the electron beam has proved too small, so it is too early to speak of such an arrangement as a practical proposition. Thermionic cathodes may be directly or indirectly heated. The emission current, i_h, is given by the Richardson-Dushman equation

$$i_h = CT^2 \exp(-A/kT)$$

where C is a constant dependent on the cathode material ($C = 4\pi mek^2/h^3$ A cm^{-2} K^{-2}), A is the electronic work function, e is the charge of an electron, and m is the mass of an electron.

An electron as it enters an electric and a magnetic field is acted upon by what is known as the Lorentz force

$$F = eE + ev H \tag{4-9}$$

where E is the electric field, H is the magnetic field, and v is the velocity of the electron.

As is seen from Eq. (4-9), an electron is imparted an energy only in the electric field; the magnetic field solely changes its direction. The force acting on an electron in a magnetic field is at right angles to the velocity of the electron. The kinetic energy that an electron acquires in an electric field is proportional to the potential difference through which it moves:

$$W = mV^2/2 = CV$$

The power of the entire electron beam striking the target is given by

$$w_1 = I_1 V_1$$

where $I_1 = qe$ is the current in the electron beam, A; q is the number of electrons in the beam passing per second; and V_1 is the accelerating voltage (actually, the difference in potential between the emitting cathode and the target), V.

When electrons strike the target, their energy is converted to heat. Like any flux of particles possessing mass and velocity, the electron beam exerts a pressure on the target. As electrons penetrate the material, they are retarded and, in doing so, they lose both kinetic energy and momentum. The change in momentum is given by

$$\Delta(mV) = mV \tag{4-10}$$

where m is the mass of an electron at rest. (Since the velocity of electrons is still far away from relativistic values, their mass is independent of velocity.)

The pressure exerted by the electron beam (in g cm^{-2}) is

$$p_e = qmV$$

Since

$$q = I_1/e = w_1/eV_1$$

it follows that

$$p_e = (w_1/eV_1)mV_1 = w_1\sqrt{2m/eV_1}$$

On substituting the constants and re-arranging Eq.(4-10), we get

$$p_e = (w_1/eV_1)mV_1 = 3.43 \times 10^4 w_1/\sqrt{V_1} = 4.37 \times 10^{-4}\pi\sqrt{V_1}/d^2$$

where $w_1 = W_1/(\pi d^2/4)$, and d is the diameter of the electron beam at the surface of the target (in cm).

Given a constant electron-beam power, the pressure exerted on the target decreases with increasing accelerating voltage. As an example, for $w_1 = 50$ kW cm^{-2} and $V = 12$ kV, the electron beam pressure is $p_e = 0.158$ g cm^{-2}.

At an accelerating potential of 5 to 100 kV, the electron penetration δ (in cm) can be found by the following equation

$$\delta = 2.1 \times 10^{-12} V_1^2/\gamma$$

where γ is the density of the target material, g cm^{-3}. As an example, at $V_1 = 20$ kV, the electron penetration will be 3.1 μm for aluminium, and 0.44 μm for tungsten.

In passing through the target material, each electron loses some of its energy in accordance with the law

$$W(x) = C_0\exp(k_x) = -d(nE)/dx$$

where E is the mean electron energy at a point, n is the number of electrons passing through unit area of the workpiece per second, k_x is the coefficient characterizing energy absorption by the target material, C_0 is a constant, and x is the distance travelled by the electron.

GLOW-DISCHARGE HEATING. In this case, the workpiece is made the cathode of a glow-discharge circuit, and the positive ions thus produced are allowed to strike the cathode. In doing so, the ions raise the spot to a very high temperature so that the cathode material evaporates or, rather, sputters. Since the cathode material has a very high thermal conductivity, the temperature of the hot spot drops very quickly, and the mean temperature of the cathode remains relatively moderate. For a glow discharge to heat the workpiece effectively, the pressure in the process chamber must be anywhere between 1.3×10^2 and 1.3×10^3 Pa. The surfaces to be joined are cleaned by cathode sputtering at a pressure of $1.3\text{-}1.3 \times 10^1$ Pa. As is seen, the bonding operation is conducted in a closely controlled atmosphere [6, 7, 13].

In sketch form, an arrangement for glow-discharge heating is shown in Fig. 4-11. The workpiece is connected as cathode, *1*, and heat is generated by an anode, *6*, fabricated from steel wire which is coiled so as to straddle the work as shown in the figure. The temperature is measured by a platinum-platinum/rhodium thermocouple, *3*, enclosed in a ceramic sleeve *5* which is placed in a thermocouple pocket *4*.

Interestingly, glow-discharge heating may utilize an inert gas, say, argon, to fill the process chamber. The chamber is first evacuated to a pressure of 1.33×10^1 Pa, then filled with argon to a pressure of about 133 Pa, and finally exhausted. This

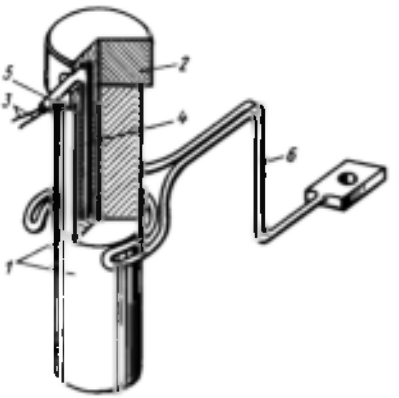


FIG. 4-11 Fixture for diffusion bonding in a glow discharge

markedly reduces the partial pressure of the residual gases, and a stable glow discharge can be maintained as the pressure is held at a constant value by admitting argon by a leak valve at a metered flow rate. A glow discharge can also be utilized to clean the surfaces before they are to be joined in vacuum. A glow-discharge unit is less expensive than an induction heating unit since there is no high-frequency current source and the operating vacuum may be as low as 1.33 Pa, which speeds up the welding operation.

INFRARED (IR) HEATING. Recent years have seen rapid expansion in the use of IR lamps which possess a number of attractive properties, such as high luminous flux density, freedom from time lag (the flux reaches 99% of its maximum value in a split second, because around 80% of energy input is emitted as radiation).

An ordinary IR lamp of Soviet manufacture is a quartz tube 10 mm in diameter and 375 mm long. It encloses a tungsten helix welded at either end to metal bases 22 mm long. The tube is filled with an inert gas (to a pressure of 812 hPa) and an amount of iodine (up to 2 mg). The function of the iodine vapour is to maintain the stability of the energy and luminous flux. The power rating of the IR lamp is 2.2 kW at 380 V. For proper operation of the tungsten filament, the lamp should be held horizontal to within 5°.

The idea to use an IR lamp as a heat source for diffusion bonding has been tried at the Paton Electric Welding Institute (USSR). The specimens were tantalum-alloy tubes laid out horizontally. Owing to this arrangement, the lamp could be placed as required for proper operation and all of the luminous flux could be utilized to advantage. The joining operation was carried out in a pressing arrangement utilizing the difference in coefficient of thermal expansion between the workpiece and the jig. The jig was placed inside a vacuum chamber which was exhausted to 1.3×10^{-2} Pa (so that the atmosphere inside the chamber could be controlled at will). The tubular tantalum-alloy specimens 25 mm in diameter and with a wall thickness of 3 mm were raised to 1223-1273 K in a matter of 1.5 or 2 min. This assured an optimal structure and good mechanical properties for the bond. Subsequent analysis revealed no difference in mechanical properties from similar specimens diffusion-bonded with h.f. induction heating.

Further advantages favouring the use of IR lamps in diffusion bonding are ease of control, a relatively long service life, and low cost.

4.6 Generation and Transfer of Pressing Load

Pressing load (bonding pressure) can be derived in several ways.

PRESSING DUE TO THERMAL INTERFERENCE OF MATING PARTS. This form of loading utilizes the interference between mating components differing in coefficient of linear expansion, when they are exposed to heat (Fig. 4-12). The resultant diffusion-bonded joint is free from residual stresses at 293 K.

The bonding variables ordinarily chosen will be clear from the example that follows. Let A be a female component of inside diameter d_A, and B be a male component of outside diameter d_B. Their respective coefficients of linear expansion are α_A and α_B such that $\alpha_A < \alpha_B$. At the time of welding the two components differ in temperature, the female part being heated to T_1, and the male part to T_2 such that $T_1 > T_2$. Since the inside diameter of the female component is then the same as the outside diameter of the male component, practically no force is needed to make the bond. Mathematically, the above relations can be written as

$$d_{A(T)} = d_{B(T)}$$

$$d_{A(0)} = d_{B(0)}$$

since

$$d_T = d_0 + d_0 \alpha \Delta T$$

it follows that

$$d_{A(0)} + d_{A(0)}\,\alpha_A T_1 = d_{B(0)} + d_{B(0)}\,\alpha_B T_2$$

and

$$\alpha_A/\alpha_B = T_2/T_1$$

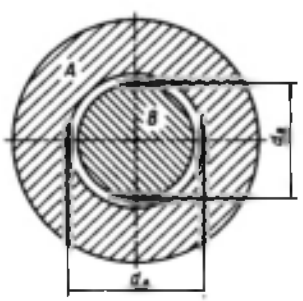


FIG. 4-12 Transfer of pressing load by thermally induced interference

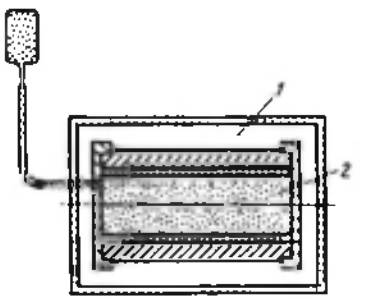


FIG. 4-13 Transfer of pressing load by gas
1 — vacuum; *2* — inert gas

The above equations serve as a basis for the choice of heating temperature required to produce thermally induced interference during diffusion bonding.

PRESSING BY GAS. Where the components to be joined are intricate in shape or have extended surfaces (such as reservoirs, heat exchangers, etc.), the necessary pressing load can advantageously be applied with a gas. The manner in which this is done will be clear from Fig. 4-13 where a foil liner is to be bonded to the inside surface of a stub pipe. The stub pipe is capped at both ends, but the cap at one end receives a connection to an inert-gas reservoir. The stub pipe is loaded into a vacuum chamber and is wound with several turns of an h.f. inductor (not shown in the figure). By metering the inflow of the inert gas, the operator can build up an optimal pressing load on the components during diffusion bonding.

PRESSING BY A PUNCH. This form of pressing is applicable when the plane of contact between the components being joined is at right angles to the motion of the punch and also when the force exerted by the punch can be resolved into two components, one being normal to the contact surface (as in joining concentric, tapering and other parts).

The load applied to the punch may come from a mechanical, pneumatic or hydraulic system. To begin with, consider a *mechanical system*. This is in effect a combination of levers. The ratio of the lever arms gives the pressing load, or bonding pressure, (in MPa) transferred to the components being joined:

$$P_L = QR_1/R_2$$

where R_1/R_2 is the arm ratio, Q is the mass of the weight in kg. To this, we should add the load due to the atmospheric pressure and subtract the force of the spring clamping the parts. Thus, the total pressure exerted on the parts being joined is

$$P_{tot} \approx P_L + 0.8D^2p_{atm} - P_s$$

where D is the diameter of the bellows in cm and P_s is the force of the spring in kg. Mechanical systems are relatively seldom used.

A *hydraulic system* has an oil tank, a filter, a safety valve, a nonreturn valve, and two lines which can be operated jointly or separately as will be explained shortly. One line applies pressure to the parts being joined (the "load" line), and the other serves to lift and lower the hood of the vacuum chamber (the "lift-and-lower" line). Basically, the system operates as follows. Oil from the tank is introduced into the system under pressure by a gear or some other pump. The leaf filter installed on the pressure line removes mechanical impurities from the passing oil which then enters under the overflow spool of the safety valve provided to protect the system against overloading. The nonreturn valve installed on the pressure line maintains the oil pressure at a constant value. The system divides into the "load" and "lift-and-lower" lines downstream from the non-return valve. Operation of the two lines is coordinated and the necessary pressure is maintained and monitored by a pressure relay and a pressure gauge installed on the "load" line upstream of the spool valve. The "load" line actuates a hydraulic cylinder whose piston transmits pressure through its connecting rod to the parts being joined. The piston and the rod are sealed with chevron seals.

When necessary, the "load" line can be isolated from the "lift-and-lower" line by a shut-off valve. When the "lift-and-lower" line is activated, oil is allowed to flow through the common elements of the system and the shut-off valve to enter the spool valve of the "lift-and-lower" system. Downstream from the spool valve, oil is admitted to a throttling valve and a regulator which smoothens the travel of the hydraulic-cylinder piston whose rod is connected to the hood of the vacuum chamber. The reversible spool valve and the oil pump are controlled by limit switches.

The system can be operated manually by push-buttons installed under a cover on the hydraulic-system console, or as part of a general automatic system. An automatic cycle consists in the following. After the workpieces are loaded into the vacuum chamber, the operator presses the "Start" button to actuate the hydraulic system. In response to the command, the pump feeds oil through the filter, safety valve, non-return valve, and shut-off valve to the spool valve which channels oil into the top space of the hydraulic cylinder. The oil pressure causes the piston to move downwards, and its connecting rod lowers the hood of the vacuum chamber. When the necessary pressing load has been achieved, the limit switch returns the spool valve to the middle position. In this position, the pressure line is cut off from drain, and the pressure is maintained at a constant value. When the parts being joined have reached the specified temperature (as sensed by a thermocouple or a photopyrometer), an electromagnet moves the spool valve to the extreme position, the pump is started, and oil is admitted to the top space of the "load" hydraulic cylinder. The line feeding the cylinder is fitted with a pressure relay installed downstream from the spool valve. When the pressure goes up or down relative to the specified value, the relay moves the spool to the opposite extreme position and stops or starts the pump. Visually, the pressure in the system can be monitored on the pressure gauge. At the end of the welding time (as indicated by a time relay) and after the workpieces have cooled to 373-473 K, the spool valve is moved to the opposite extreme position in which oil is allowed to lift the piston. In its topmost position the piston actuates the limit switch, and this moves the spool valve to the middle and neutral position and actuates a second spool valve in the "load-and-lift" line. In the topmost position the limit switch de-energizes the solenoids, thereby moving the spool valve to the middle position and turns off the pump. This completes the cycle which is then repeated all over again.

4.7 Classification of Vacuum Diffusion Bonding Equipment

In the Soviet Union, over 70 designs of equipment for diffusion bonding in vacuum have been developed at this writing. They are usually classed as proposed in [6] into the types shown in Fig. 4-14.

The key components of a vacuum diffusion bonding unit or machine are a vacuum bonding chamber, a vacuum pumping system, a source of heat, and a pressing load system. It usually comes complete with assorted accessories, such as pressure and vacuum gauges, thermocouples, etc. The units are usually arranged along the same lines as welding machines using other processes. It appears that the

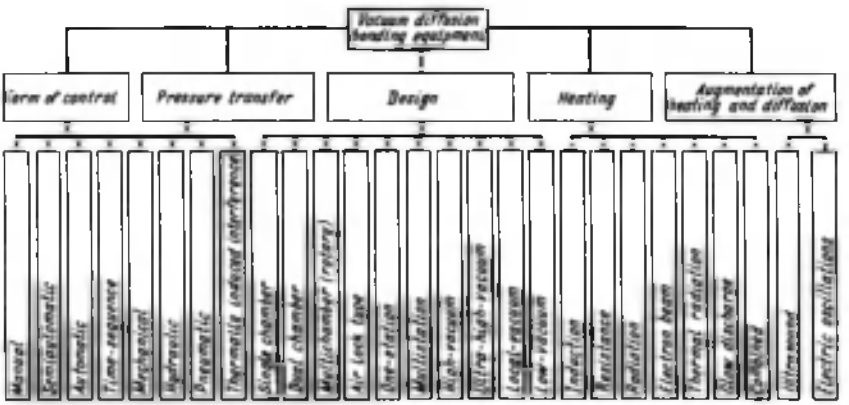


FIG 4-14 Classification of vacuum diffusion bonding equipment

designers of vacuum diffusion bonding equipment have as yet failed to utilize all of the potentialities offered by the vacuum diffusion bonding process.

According to scale of fabrication, vacuum diffusion bonding machines can be classed into those adapted to piecework, batch work, and mass-scale work. Those in the last two groups may be rotary or conveyer-type automatic units.

According to the vacuum used, vacuum diffusion bonding machines may be classed into low-vacuum units (operating at 1.3×10^{-1} Pa or less), medium-vacuum units (operating at 1.3×10^{-1}-1.3×10^{-3} Pa), high-vacuum units (operating at over 1.3×10^{-3} Pa), and those in which the shielding gas is used at a reduced or an elevated pressure.

The workpieces may be placed in vacuum completely or partly (locally). In the latter case, bulky pieces (bars, pipes, etc.) are joined with vacuum applied around the joint so as to protect it against exposure to the atmosphere.

Heat for vacuum diffusion bonding can be supplied by any known sources (radiation, induction, plasma, arc, light beam, etc.), the choice being made according to the bonding temperature needed, the design and shape of the work. Accordingly, diffusion-bonding machines may be classed into induction types, radiation types, resistance types, electron-beam types, laser types, glow-discharge types, or combination types. The most commonly used are induction-type machines which owe their popularity to their simplicity and adaptability to a widely varying range of products. Resistance heating is warranted in cases where heat should be localized within the joint area. Radiation heating is recommended for joining thin-walled pieces and nonmetals. Heating by thermal conduction is employed in the diffusion bonding of dissimilar materials. The glow discharge is used in cases where it is important to cut down the bonding time (because glow-discharge units do the cleaning and heating of the workpieces at the same time). Electron-beam and infrared heating from quartz tubes comes in handy in joining refractory metals (such as tungsten, molybdenum and zirconium).

The manner in which the pressing load (bonding pressure) can be applied to the workpieces depends on their configuration, with the punch moving at right or some other angles to the contact area. The necessary pressure can be derived hydraulically, pneumatically, mechanically, thermally, electrically, with the piston rod made to dither about the plane of the joint, with the superposition of ultrasonic vibrations, an electrostatic and magnetic field, etc. The most commonly used types are those with hydraulic and mechanical pressing systems. Pressing due to thermally induced interference can utilize ordinary vacuum or gas furnaces.

Vacuum diffusion bonding machines may have one or several vacuum chambers. Multichamber units have a higher throughput against a relatively small increase in the floor area required. Also, the heat source can be better utilized. As a rule, the maximum number of chambers per unit is two to three.

Within the vacuum chamber, the workpieces may take up one or several stations or positions. Multistation units can join several articles at a time, so the scale of fabrication is increased in the same proportion. The optimal number of stations is two to four; a greater number will usually lead to nonuniform heating.

Still another way to class vacuum diffusion bonding machines is into those manually operated, semiautomatic, and programmed automatic units. Most commonly, use is made of manually operated and semiautomatic machines. They may be stationary and mobile.

Vacuum diffusion bonding machines may be general-purpose and special-purpose. General-purpose machines were predominant in the early years of diffusion bonding when little was known about the potentialities and limitations of the process. With further advances in the field, machines have appeared, specifically adapted for the fabrication of electron tubes, semiconductors devices, instruments, etc.

Vacuum diffusion bonding machines can operate intermittently or semicontinuously. The latter include rotor and conveyer-type units.

Current work in the USSR has as its objective to develop unified and standardized vacuum, heating, pressing and control systems and process chambers. By using a range of process chambers one and the same machine can be adapted to handle a wide range of products differing in design and size.

The choice of a particular type of machine is decided by the desired output, the design and shape of workpieces, and the properties of the materials that will be handled.

REQUIREMENTS TO BE MET BY A VACUUM DIFFUSION BONDING MACHINE. Diffusion bonding machines use a dynamic vacuum pumping system. This implies that the system is operating continuously because the workpieces being heated and joined give out gases and vapours. It goes without saying that all the elements of the vacuum system (the pumping facilities, valves, lines, and instruments) should be completely vacuum-tight. Also, it should maintain the specified pumping speed, its lines (including the main line of the chamber) should present a minimum resistance, and its pumps should have an ample capacity. The vacuum system must be convenient to operate and service and take up as little floor area as practicable. All this goes a long way towards producing sound joints by diffusion bonding in vacuum.

Obviously, with a poor vacuum in the process chamber there would be a large amount of water vapour, oxygen and other gases which would react with the materials of the workpieces, thereby impairing the strength of the joints.

For a diffusion bond to be strong and sound, the vacuum in the process chamber must be anywhere between 1.3 and 1.3×10^{-5} Pa, depending on the material and properties of the pieces being joined. Of course, this wide range of vacuum cannot be produced by any one method. At present, this is done by two basic methods, namely (a) by means of vacuum pumps and (b) by means of getters which absorb the oil and water vapours present in the vacuum system and thus improve the vacuum. There is also an auxiliary method involving the use of cryogenic traps. The vacuum ranges for which various vacuum pumps, getters and traps are best suited are described in detail in the literature [3, 4, 5, 7, 10].

The rules for the installation of vacuum equipment on a vacuum diffusion bonding machine may be summed up as follows.

(1) The high-vacuum pipework should have the largest possible diameter and the shortest length (the diameter of the pipework should be no less than that of the associated oil-vapour pump).

(2) Each diffusion or booster oil-vapour pump should be fitted with an oil baffle.

(3) The size of the forevacuum (roughing) pump should be chosen to suit the counterpressure and throughput of the associated diffusion or booster oil-vapour pump. The recommended forevacuum pumps of Soviet manufacture are listed in Table 4-15.

(4) The diameter of the roughing line should be chosen equal to that of the receiving port on the roughing pump. After the length of the vacuum line is determined, the system should be analyzed for the pressure drop in the pipework so as to make sure the pressure downstream of the oil-vapour pump is below critical.

(5) Compensators should be installed between the roughing pumps and the remaining vacuum system and also where closed loops are formed.

TABLE 4-15

Oil-vapour booster or diffusion pump	Recommended roughing pump
BN-3: S = 500 at 0.665 Pa	VN-1G: S = 14 at 39.99 Pa
BN-1500M: S = 1500 at 1.333 Pa	VN-4G: S = 40 at 199.96 Pa
BN-4500: S = 4500 at 1.333 Pa	VN-6G: S = 110 at 133.3 Pa
N-1S: S = 100 at 0.026664 Pa	VN-461M: S = 0.3 at 0.99975 Pa
N-5S: S = 500 at 0.026664 Pa	VN-2G: S = 5 at 0.99975 Pa
N-2T: S = 2000 at 0.026664 Pa	VN-2G: S = 5 at 13.3 Pa
N-5: S = 4500 at 0.026664 Pa	VN-1G: S = 14 at 13.3 Pa
N-8T: S = 8000 at 0.026664 Pa	VN-11: S = 14 at 13.3 Pa

Note. S – pump speed, $l\ s^{-1}$.

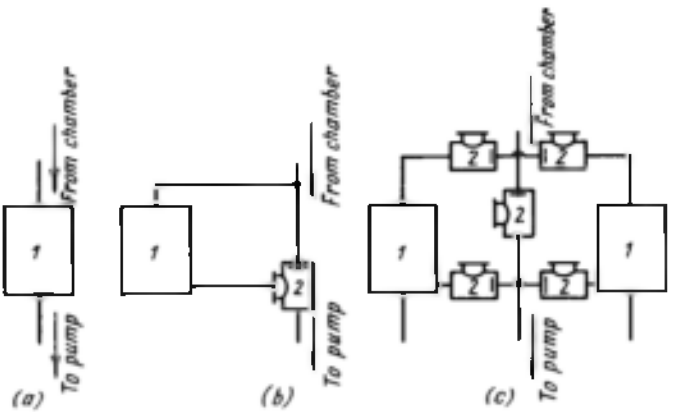


FIG. 4-15 Location of filters and traps
1 — filter or trap; *2* — valve

(6) Filters and traps should be arranged in any one of the layouts shown in Fig. 4-15. Layout (*a*) should preferably be used in cases where the exhausted gas must be cleaned during the operation of an intermittently operating diffusion bonding machine. If gas cleaning is required only at the start of the pump-down (which accounts only for a fraction of the total joining time), preference should be given to layouts (*b*) and (*c*). In these layouts, the filter or the trap having a high resistance can be brought out of the system with a shut-off valve, and the pump-down will then proceed along the line having least resistance. Layout (*c*) should be used in continuously and semicontinuously operating machines, so that the filter or the trap can be cleaned out while the machine is running.

(7) The vacuum flap valves must be located so as to permit testing of any vacuum pump closed on itself, and also transfer the vacuum chamber between several types of pump.

(8) The sensing elements of the vacuum gauges must be located close to the pumped vessel and at the pumps so that the latter can conveniently be tested for operation with its ports closed.

(9) An admission valve should be provided so that air or a shielding gas can be leaked in whenever necessary. This valve should be located close to the roughing pump and adapted to receive a helium leak detector.

(10) Non-purgable nitrogen traps of low throughput should be installed only when ingress of oil vapour into the pumped chamber is highly objectionable.

Typically, a vacuum diffusion bonding machine incorporates a mechanical roughing pump and an oil-vapour booster or diffusion pump.

COMPONENTS AND LAYOUT OF THE VACUUM SYSTEM. The valves and lines for a vacuum system should be chosen to suit the requirements as regards the ultimate pressure (vacuum in the chamber) and the degree of automation employed. The pumping facilities should be chosen so as to ensure the desired ultimate pressure and

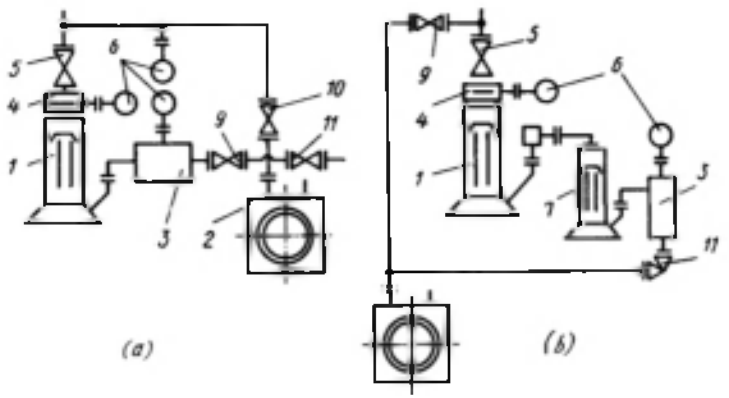


FIG. 4-16 Layouts of high-vacuum systems

to handle the gas load (the gas entering the pump from the pumped vessel). The instrumentation (vacuum gauges and their accessories) should suit the vacuum range used in the system. Two layouts for vacuum systems employed in vacuum diffusion bonding machines in the pressure range 1.3×10^{-2}-6.7×10^{-5} Pa are shown in Fig. 4-16. In layout (*a*) the system produces an ultimate pressure of 4.2×10^{-5}-6.7×10^{-5} Pa and consists of a vapour diffusion pump *1*, fitted with nitrogen traps *4*, and a mechanical vacuum pump *2*. The further components of the system are a high-vacuum valve *5* on the high-vacuum side, a bypass pumping line fitted with a valve *9*, a forevacuum tank *3*, and emergency shut-off valves *8*. The high-vacuum valve isolates the vacuum system from exposure to the atmosphere when a new workpiece is loaded in the chamber. The bypass pumping line and valve make it possible to avoid any waste of time that would otherwise be necessary in order to cool and warm up the vapour diffusion pump. When air is being exhausted from the process chamber via the bypass line, the vapour diffusion pump is discharging into the forevacuum tank which is isolated at that time from the mechanical pump by the emergency shut-off valve. The shut-off valve will prevent ingress of air and oil from the mechanical pump into the vapour diffusion pump, should a power cut occur in the system. Sometimes, one more valve, *10*, can be installed so as to prevent ingress of oil from the mechanical pump into the shut-off valve, *8*, and the bypass valve, *9*. To this end, when the system is stopped, the space above the inlet port of the mechanical pump is opened to the atmosphere. Pressure at various points of the vacuum system is measured by vacuum gauges, *6*.

In layout (*b*), the vacuum system contains a vapour diffusion pump *1*, a vapour booster pump *7*, a mechanical vacuum pump *2*, and an oil baffle *4* installed in the inlet port of the vapour diffusion pump. Provision of the booster pump between the vapour diffusion pump and the mechanical vacuum pump enables heavy gas loads (heavy outgassing) to be handled and a sufficiently low ultimate pressure to be obtained. Practically, the system can produce an ultimate pressure of

4.2×10^{-5}-6.7×10^{-5} Pa. An important advantage of the system is that the working fluid of the vapour diffusion pump is well degassed. The process chamber is exhausted to a high vacuum via a valve *5*, whereas the bypass pump-down is effected by the mechanical pump via valve *9*. During the bypass pump-down the angle valve *11* is closed, and the booster pump discharges into the forevacuum tank *3*. Pressure is measured by vacuum gauges *6*.

The requirements for tightness and outgassing in the case of systems operating at pressures below 6.7×10^{-5} Pa are more stringent than they are for systems operating above 6.7×10^{-5} Pa. Among other things, such systems may not use rubber gaskets and all the components must be baked to 573-873 K for better degassing. The materials that go to make such systems should have a low vapour pressure at the degassing (bake-out) temperature, be readily weldable, have a sufficient mechanical strength, and resist corrosion well. In the Soviet Union, the components for such ultra-high-vacuum systems are fabricated from 12Х18Н10Т steel. Permanent joints are made with the argon-arc or electron-beam welding process. Permanent joints are usually sealed with copper gaskets. Two likely layouts for the ultra-high-vacuum system of a vacuum diffusion bonding machine are shown in Fig. 4-17. In layout (*a*), the very high vacuum is produced by a hot-cathode getter-ion or a cold-cathode getter-ion pump, *1*, which evacuates the process chamber through an all-metal bakeable valve *2*. There is also a vapour diffusion pump, *5*, fitted with a nitrogen trap, *4*, which serve to pre-degas the getter-ion pump and the vacuum system and to rough down the chamber. The pump-down to 1.3 Pa is effected by a mechanical vacuum pump, *6*, through the vapour diffusion pump, *5*, while it is cold. The vapour diffusion pump can be isolated from the rest of the vacuum system with an all-metal bakeable valve *3*. The exhaust port of the vapour diffusion pump is connected to the mechanical vacuum pump via a valve *9*. Pressure is measured by vacuum gauges *7* and *8*.

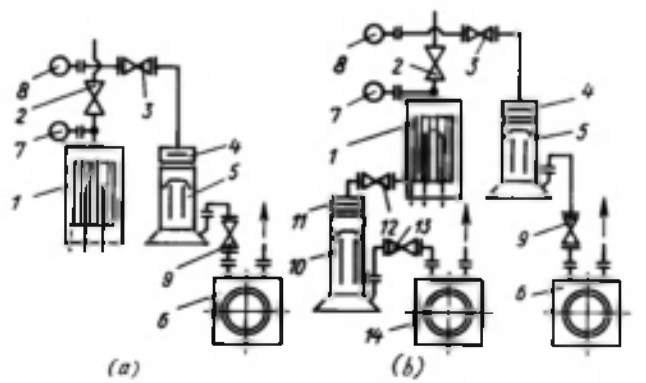


FIG 4-17 Layouts of ultra-high vacuum systems

In layout (*b*), the vapour diffusion pump *10* is fitted with a nitrogen trap *11* and a bakeable valve *12*. Addition of the vapour diffusion pump, *10*, to the getter-ion pump, *1*, substantially raises the speed of exhaust at low pressure, as it can pump out inert gases. The vapour diffusion pump, *10*, is backed by a separate mechanical vacuum pump, *14*, which is connected by a pipework via a valve *13*. The vapour diffusion pump, *10*, can be isolated from the rest of the vacuum system by an all-metal valve *12*.

Figure 4-18 shows the arrangement of a vacuum system that can typically be used in machines adapted to diffusion-bond large articles. The process chamber *5* is exhausted by a turbomolecular pump *1* via a bakeable valve *3*. In turn, the turbomolecular pump is evacuated via a valve *11* by a mechanical vacuum pump *10*. When the machine is stopped, the vacuum system is filled with dry nitrogen via a valve *2*. Because it has a large volume, the process chamber is roughed down via a valve *7* by a Roots blower pump *8* which discharges against another mechanical pump, *9*. Each pump is built as a stand-alone unit. When the process chamber is roughed down and degassed, it is filled with inert gas via valve *6*. Pressure in the system is measured with a vacuum gauge *4*.

The capacity of a vacuum diffusion bonding machine can be substantially enhanced by arranging it into a rotary type. With a rotary machine, less time is expended on auxiliary operations and a greater number of process chambers can be used. In fact, the increased number of process chambers is a necessity, because a straightforward increase in the operating speed of the machine would degrade the degassing of the workpieces. A further advantage is that the finished product is removed and a new workpiece is mounted at one and the same position. The pump-down stations of a rotary machine can use independent vacuum systems which are built so that only some part of the system is turning along with the process chamber while the remaining part is held stationary. Communication between the movable and stationary parts of the system is by means of a spool valve which also acts as a multiway vacuum valve. This arrangement markedly simplifies the design of rotary

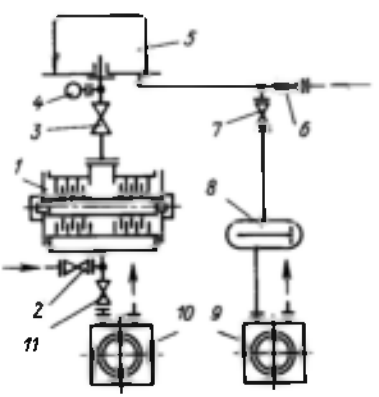


FIG. 4-18 Layout of a vacuum system for the diffusion bonding of large pieces

machines and control of their vacuum systems; also it permits a substantial reduction in the weight and size of the rotating parts. The spool valve may be located on the high- or low-vacuum side. Unfortunately, spool valves lack in tightness, and this, coupled with the omission of vacuum lines, serves to raise the pressure downstream from the spool valve over that existing at the outlet port of the associated vacuum pump.

Two likely layouts of vacuum systems for rotary diffusion bonding machines are shown in Fig. 4-19. In layout (*a*), the system has no spool valve; valves *3* through *6* are combined into a single unit so that valves *4*, *5* and *6* are actuated by a common mechanism which has two operating positions. In one position, valves *4* and *6* are closed, and the system is roughed down by a mechanical pump *2* via valves *3* and *5*. In the other position, valve *5* is closed, and the process chambers are exhausted to a high vacuum by a vapour diffusion pump *1* via valves *3* and *4*. At the same time, the outlet port of pump *1* communicates via the open valve *6* with the mechanical pump *2*. When a new workpiece is to be loaded, valve *3* is closed. A total of 24 pumping stations arranged as just described can be set up on a single rotary machine.

In layout (*b*), the system serves 16 work stations divided into four groups each served by a multi-chamber mechanical vacuum pump *7* set up in the lower part of the rotary frame. After a finished product has been removed and a new workpiece

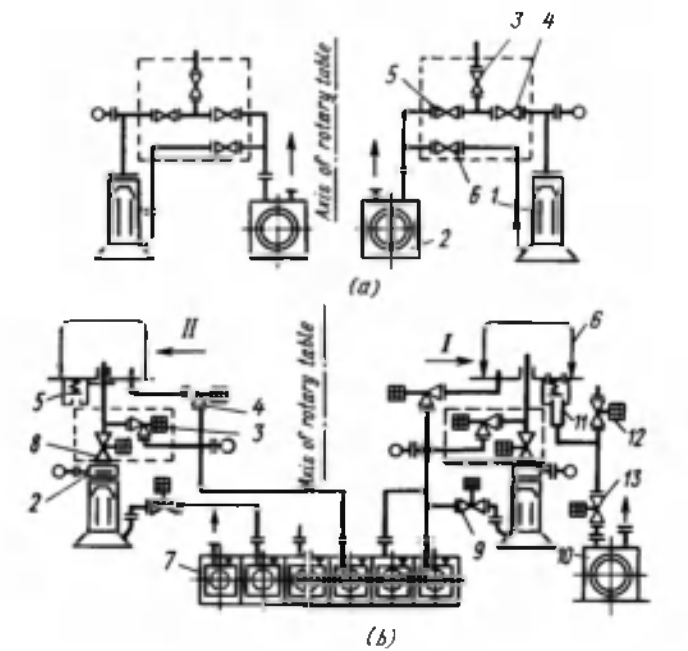


FIG 4-19 Layout of vacuum systems for rotary-type diffusion bonding units

loaded (station *I*), the process chamber *6* is roughed down by a stationary mechanical vacuum pump *10* via a solenoid-operated valve *13* and a valve *5* actuated by a mechanical drive *11*. Atmospheric air is admitted to chamber *6* via a solenoid-operated valve *12* and valve *5*, while valve *13* is closed. As the rotary frame is advanced, drive *11* isolates valve *5* from the stationary vacuum system (which exhausts the process chamber to a rough vacuum or admits air to the chamber). The stationary mechanical pump improves the roughing-down of the process chamber and facilitates the operation of the multichamber mechanical pump *7* as it finally exhausts the process chamber (station II). The pump-down of the process chambers to the ultimate pressure is effected via a solenoid-operated valve *8* by a vapour diffusion pump *1* fitted with an oil baffle *2*. Solenoid-operated valves *3* and *8* are built into a common valve body. The outlet port of pump *1* is connected via an emergency solenoid-operated shut-off valve *9* to one of the chambers in the multichamber mechanical pump *7*. The basic purpose of valve *9* is to prevent ingress of atmospheric air and oil from pump *7* into pump *1*, should a power cut occur in the system.

Further arrangements of vacuum systems are shown in Fig. 4-20. In layout (*a*), the process chamber is exhausted in three steps. At first it is pumped to a rough vacuum by a mechanical pump *3* (station *I*), then roughing-down is continued by a multichamber mechanical pump *2*, and the pump-down to high vacuum is effected by a vapour diffusion pump *1* (station *II*). In this layout, the spool valve *4* is located between the process chamber and the vapour diffusion pump. For reliable operation, the vacuum passages of the spool valve are protected by annular grooves filled with oil which is exhausted by pump *2*. Systems with this type of spool valve can give an ultimate pressure of not better than 1.3×10^{-7} Pa, no matter how perfect the system elements are. This occurs because of the large length of the high-vacuum lines and low tightness of the spool valve. The process chamber is loaded and unloaded when the spool valve is in position *III*.

In layout (*b*), both the process chamber and pump *1* are evacuated to a rough vacuum by a separate mechanical pump *3* (station *I*). Further pump-out of vapour diffusion pump *1* is effected by a multichamber pump *2* (station *II*). The vapour diffusion pump is periodically cooled before removal of the finished product and loading of a new workpiece (station *III*). The spool valve *4* is located in the rough-vacuum part, owing to which the system can be exhausted to a better ultimate pressure than in the previous case. Layout (*b*) is usually employed in rotary-type machines in which the processing cycle is long enough for the vapour diffusion pump to be cooled at one or two stations.

Of course, the layouts discussed above do not exhaust all the likely arrangements of vacuum systems applicable in rotary diffusion bonding machines.

INDUSTRIAL DIFFUSION BONDING MACHINES. These are intended for use in various industries. Their basic components (such as the vacuum system, the pressing system, and the heat source) and the necessary instrumentation are manufactured in the Soviet Union in quantity. The only steps involved in setting up a machine are to fabricate the process chamber, to set up the cooling and control systems, and to assemble the machine.

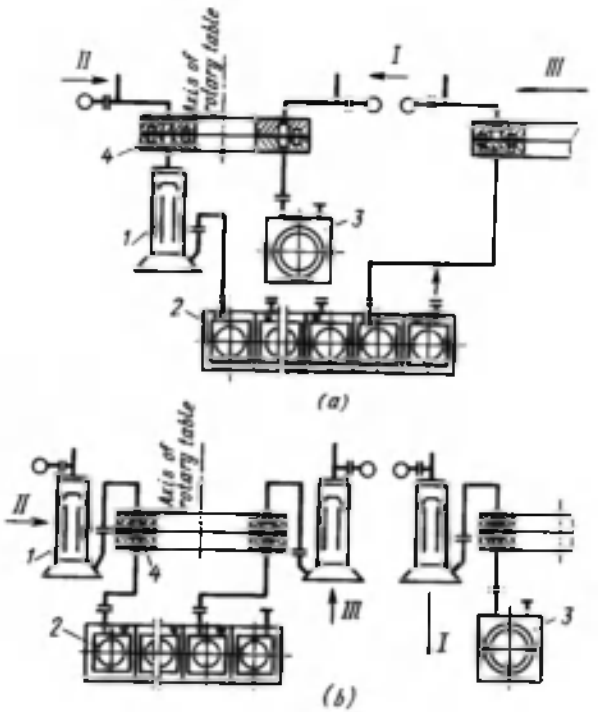


FIG. 4-20 Layouts of vacuum systems for chamber exhaustion in three steps

In the Soviet Union, several types of industrial diffusion bonding machines are available. We shall dwell in brief on some of them.

Induction-heating machines. This is the most commonly used form in industry and research. At the time of writing, about 40 types were in service. Of their total, about 90% are general-purpose, and about 10% are special-purpose.

The type SDVU-50 machine (Fig. 4-21) can diffusion-bond various articles (up to 40 cm long and 20 cm in diameter) from similar and dissimilar metals and nonmetals. The machine is assembled from unified components and has one process chamber in which the workpieces are induction-heated. Pressing load is transmitted electromechanically, and the machine is operated remotely. It is designed for installation in a dry ventilated location with an ambient temperature of 298 ± 10 K at a relative humidity of $65 \pm 15\%$ and a pressure of 100 ± 4 kPa, in an atmosphere free from acid and alkali fumes or vapours and other corrosive substances. Heat for diffusion bonding is supplied by a 60-kW r.f. valve oscillator. The maximum pressing load is 100 kN. The maximum heating temperature is 1 773 K. The operating vacuum is 6.7×10^{-3} Pa. The machine measures $1.1 \times 0.72 \times 3.5$ m overall and weighs 520 kg.

FIG. 4-21 SDVU-50 industrial diffusion bonding unit

The diffusion-bonding chamber of the machine is fabricated from a corrosion-resistant steel and has a loading port which is covered by a door fitted with swung-out hold-downs. The progress of diffusion bonding can be watched through an observation port in the chamber door.

The type SZhM2-327 unit (Fig. 4-22) can diffusion-bond in vacuum a variety of components from similar and dissimilar metals and nonmetals at a welding temperature of up to 1 373 K. Heat for diffusion bonding is supplied by a 25-kW r.f. valve oscillator. The pressing load is supplied by a hydraulic system in two ranges, namely 500-10000 N and 10000-100000 N. The machine measures $1.5 \times 1.0 \times 2.1$ m overall and can perform two operating cycles every hour. The stationary top part of the process chamber is a cylinder welded from a corrosion-resistant steel. The lower movable part is likewise welded from a stainless steel. The joint between the two parts is sealed by a vacuum-rubber gasket. Watch on the progress of diffusion bonding can be kept through an observation window screened with a metal gauze. The bonding pressure is transmitted by a rod sealed with a bellows and copper gasket clamped between flanges. The bellows permits the rod to move

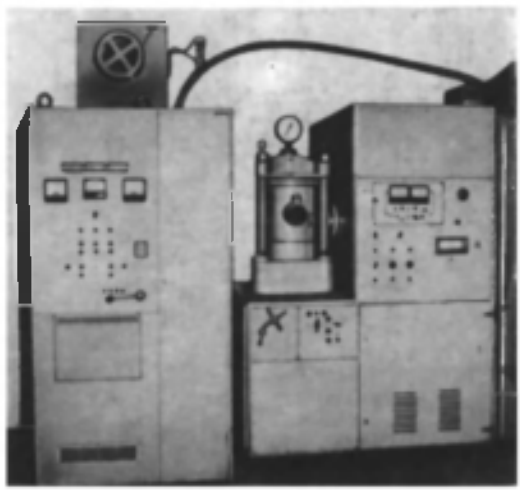


FIG. 4-22 SZhM2-327 diffusion-bonding unit

through up to 0.01 m. The load applied to the workpieces is transferred by a water-cooled punch built into the chamber lid. The inductor lead-in is located back of the chamber which is illuminated in the inside.

The type SDVU-30L machine is designed for the vacuum diffusion bonding of large metal and nonmetallic components. Heat is supplied by a 100-kW r.f. valve oscillator. The chambers of the machine operate cyclically. The hydraulic system can supply a maximum pressing load of 700 000 N. The maximum temperature to which the workpieces can be heated is 1 773 K. The overall dimensions of the machine are $4.25 \times 1.2 \times 3.35$ m, and its weight is 3 500 kg.

The type SDVU-24 unit (Fig. 4-23) is designed to diffusion-bond in vacuum small components of similar and dissimilar metals and nonmetals. The maximum size of the workpieces is 0.04×0.1 m. The maximum temperature attained by the workpieces is 1 773 K. The mechanical pressing system supplies a bonding force of 10 kN or a bonding pressure of 2-7 MPa as measured at the surface of the workpiece.

The general-purpose dual-chamber type SDVU-15-2 machine is intended to diffusion-bond articles up to 0.25 m in diameter, fabricated from various metals and alloys that can be heated to a temperature of not over 1 573 K. Heat is supplied by a 100-kW r.f. valve oscillator. Each chamber has an exhaust system of its own, capable of producing an ultimate pressure of down to 1.3×10^{-2} Pa. The hydraulic system which, like the pump-down system, is built into the machine frame provides for operation of each hydraulic cylinder independently of the other. The maximum pressure that can be built up by the hydraulic pump is 1 000 MPa. At this pressure

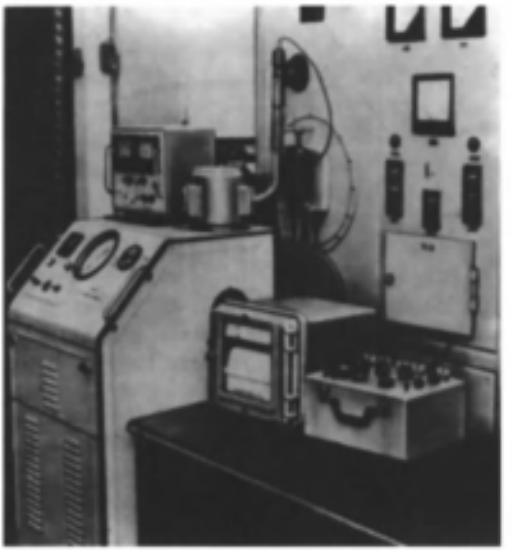


FIG. 4-23 SDVU-24 diffusion bonding unit for small parts

in the system, the hydraulic cylinders can develop a pressing load of up to 210 kN. The overall dimensions of the machine are 3.1 × 1.6 × 2.2 m. For higher output, provisions are made for diffusion-bonding of two to four components in each chamber at a time. The machine has proved its efficiency and reliability in service [7, 8].

The dual-chamber type UDS-3 machine is intended to vacuum-bond various components from metals, alloys, and nonmetals with a cross section of up to 2×10^{-3} m^2 and a height of not over 0.1 m. The two chambers mounted on the machine frame measure 0.28 × 0.28 m each and are evacuated by a separate system to an ultimate pressure of 6.7×10^{-2} Pa. The machine has two lever-type pressing systems which can develop a pressing load of 200-10000 N. Heat for bonding is supplied by a 60-kW r.f. valve oscillator. The maximum temperature to which the workpieces are heated does not exceed 1 773 K. The overall dimensions of the machine are 1.04 × 2.20 × 1.95 m.

The type UDS-4 machine is specifically designed to join hard-faced punches. It consists of the same systems (pump-out, vacuum gauging, pressing, heating and cooling) as any other machine. Its control system provides for manual operation and automatic control.

Figure 4-24 shows a machine adapted from the type SDVU-17 and intended for the diffusion-bonding of cermets. It consists of a frame *2*, uprights *3*, a quartz tube *11*, and an inductor *9* energized from a 60-kW r.f. oscillator. Uprights *3* are free to move in the guides *1* of the baseframe, so that a quartz tube of any length can be made fast on the guide with ordinary bolted joints. The cermet bushings to be joined are strung (like beads) on a bar *10* and clamped together with limit nuts *8* and *12* (for ease of centring). The stack thus assembled is loaded in the quartz tube and

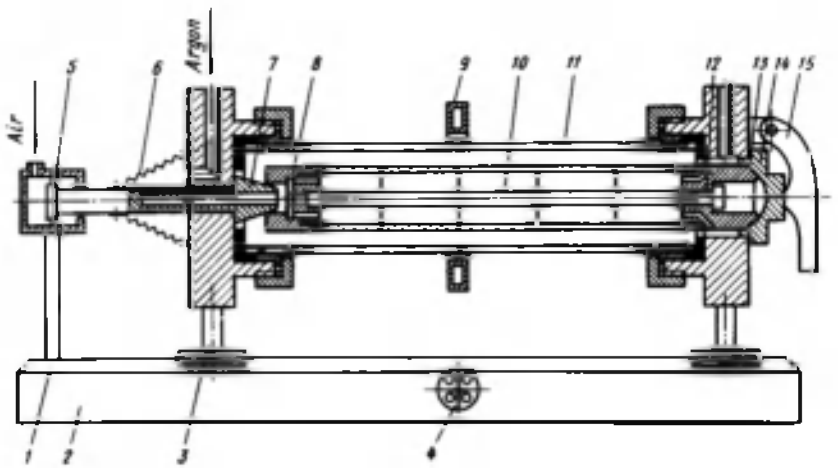


FIG. 4-24 Diffusion-bonding unit for joining cermet parts

clamped up against a pusher *7*. One end of the cermet assembly is held by the spherical part of a limit-stop *12* in the lid *14*. The lid is gasketed by a rubber ring *13* and held down by an eccentric *15*. At the other end of the quartz tube is a bellows valve *6* to clamp the parts being bonded. After the workpieces are loaded for bonding, compressed air is introduced into a pneumatic chamber *5* via a pressure regulator with which the air pressure can be maintained at anywhere between 0 and 0.6 MPa. The air acts on the pusher, and this transmits pressing load to the cermet parts. The pressing load is maintained constant throughout the bonding operation. The joint area is heated by the single-turn r.f. inductor *9* put on the outside of the quartz tube. After one joint is completed, uprights *3* are moved to the next with the feed wheel *4*.

The type SDVU-21 machine (Fig. 4-25) is designed to diffusion-bond wire 0.003-0.006 m in diameter of any length, made of various materials. The machine produces only local vacuum, that is, around the joint. The ultimate pressure achieved in the machine is 1.3×10^{-2} Pa. All clamps and locks are actuated by various hand-operated mechanisms [6-8]. In other respects, the machine is similar to those already discussed.

The type SDVU-13 machine (Fig. 4-26) is adapted to diffusion-bond active metals in vacuum. Since active metals form oxides almost instantaneously, they are ordinarily joined with difficulty. In the machine, the problem is solved by removing oxides with ultrasound. To this end, magnetostriction transducers *3* and *4* and sonotrodes *1* and *2* are applied to the two pieces to be joined. The oxide film breaks down and is removed from the mating surfaces. During the subsequent operation ultrasound applied at right angles to the mating surfaces speeds up diffusion, and this improves the strength and quality of the bond.

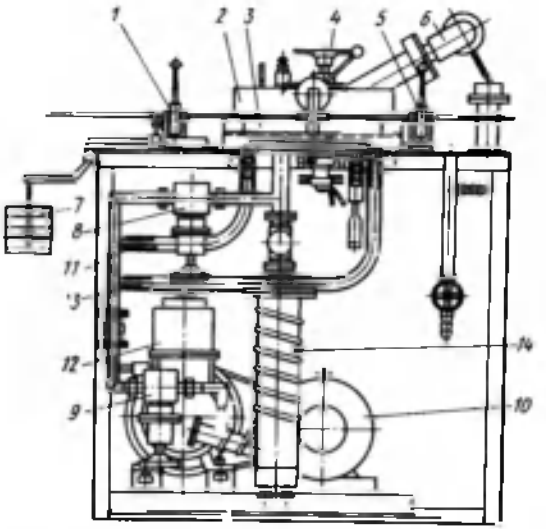


FIG. 4-25 SDVU-21 unit with local vacuum

1 — movable wire clamp; *2* — top lid of chamber; *3* — bottom lid of chamber; *4* — split inductor; *5* — stationary wire clamp; *6* — photopyrometer; *7* — weight; *8*, *9* — vacuum valves; *10* — electric motor; *11* — water dispenser; *12* — forevacuum pump; *13* — vacuum lines; *14* — high-vacuum oil-vapour pump

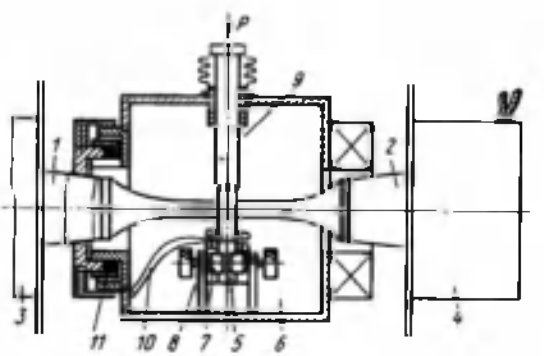


FIG. 4-26 SDVU-13 unit utilizing ultrasound

A diffusion-bonding machine developed in Poland [8] can join parts in vacuum, in inert gases, and in a shielding atmosphere. Its pneumatic pressing system can apply a bonding force of 10 kN. The parts that can be bonded in the machine may be up to 0.05 m in diameter and up to 0.1 m in height. The maximum temperature to which the parts can be heated is 1 273 K. The machine consists essentially of a vacuum chamber, a vacuum system, an inert-gas cylinder, a bonding-force transfer system, a heating and cooling system, a control system, and instrumentation.

Electron-beam machines. An example of this class of machines is the type A306-14 designed to vacuum-bond components for electron devices from refractory metals and alloys and some structural steels [11, 12]. The machine (Fig. 4-27) consists of a joining section and a power supply section. The joining section has a vacuum chamber *10*, an exhaust system, a cooling system, a hydraulic system, and three electron-optical systems *2*. The vacuum chamber is a cylinder 0.49 m in diameter and 0.48 m high, enclosed in a water jacket and it can accommodate workpieces up to 0.12 m in diameter and up to 0.18 m high. The electron-optical systems are set up on a cooled pedestal at the bottom of the chamber. The pedestal also receives the work holder *1*. The chamber is connected to a high-vacuum exhaust unit. The hydraulic system which transfers the bonding force to the workpieces has a hydraulic cylinder set up on top of the vacuum chamber and enclosing a two-step piston which can develop pressing load in two ranges, 500-10000 N and 10000-100000 N. The electron-optical systems are arranged outside the process chamber *10* in a horizontal plane and are spaced 120° apart. Attachment of the systems to the chamber sides is by flanges. Power for the electron-optical systems is supplied by the 20-kW, 10-kV power supply unit. At an accelerating voltage of 10 kV, the total electron-beam current is 2 A and the total power consumption of the machine is not over 32 kW. The power-supply unit consists of an H. V. rectifier,

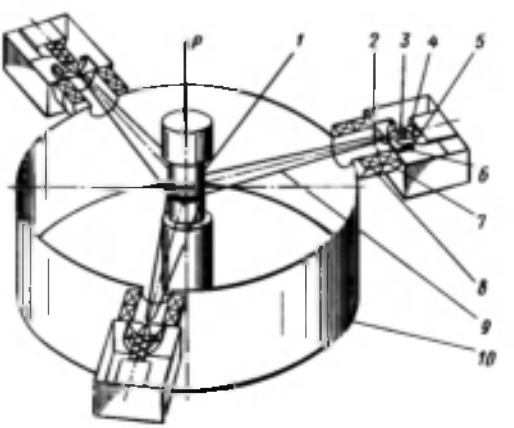


FIG. 4-27 Electron-beam heat source for A306-14 diffusion-bonding unit

a control section which maintains the voltage applied to the control electrodes *5* of the electron guns anywhere from 0 to 2 kV, a supply section for the focusing (*7*) and deflecting (*8*) coils which control the electron beams *9* and the level of heating. The deflecting coils are energized with a direct current and an alternating current at commercial frequency, so that the electron beam can be scanned across the joint being made. The focusing coils control the width of the heated zone.

The power-supply section has provision for the workpieces to be heated to a predetermined time sequence and to a predetermined temperature. Provisions are also made for diffusion bonding to be carried out on a semiautomatic basis.

The control section of the power-supply unit has a bias circuit to apply control voltage to the 10-kV rectifier, another bias voltage to supply control voltage to the control electrodes *5* which regulate the level of heating, and a supply section feeding the deflecting and focusing coils. The temperature of the workpieces is monitored and controlled within predetermined limits by a potentiometric controller. The heating time in the automatic operation is maintained by a time relay. The electric system of the machine also includes elements belonging to the exhaust and hydraulic systems, and a control panel for the machine as a whole. The electron guns are properly shielded to avoid X-ray hazard. The overall dimensions of the machine are 1.78 × 1.73 × 2.1 m.

Radiation-heating machines. This class is exemplified by the type SDVU-38M which is intended to diffusion-bond workpieces measuring up to 0.04 × 0.1 m at a maximum temperature of 1 273 K and a bonding force of 2 kN which is supplied by an electromechanical system. The workpieces are heated by radiation emitted by a

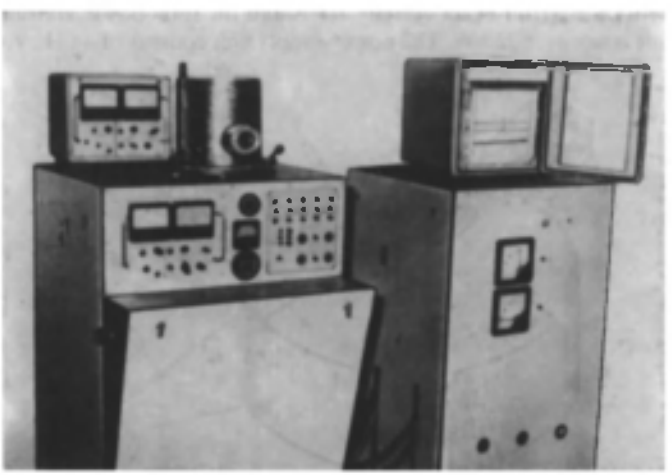


FIG. 4-28 SDVU-60 unit utilizing radiation heating

tantalum foil or tungsten rods. The optimal ultimate pressure in the bonding chamber is 6.7×10^{-2} Pa. The machine can be operated manually and automatically.

The type SDVU-60 machine (Fig. 4-28) is intended for research into diffusion bonds made at a vacuum of 6.7×10^{-3} Pa in various nonmetals (ceramics, glass, pyrosil, and semiconductors). It has a cylindrical process chamber 0.32 m in diameter and 0.3 m high. The workpieces are heated by a cylindrical radiator fabricated from molybdenum ribbon which carries electric current. The maximum temperature of the workpieces is 1 473 K. The maximum dimensions of the workpieces that can be accommodated in the chamber are: diameter, 0.1 m; height 0.1 m. The bonding force is transferred to the workpieces by an electromechanical drive. The maximum bonding force is 5 kN, as adjusted for atmospheric pressure.

The type A308-13 machine is designed for the precision diffusion bonding of microminiature ceramic components to metal parts in vacuum and a gas at-

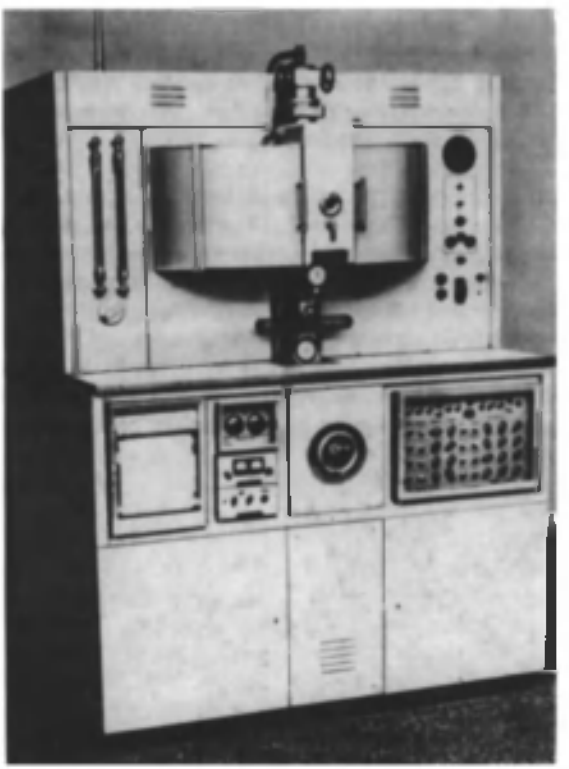


FIG 4-29 A306-21 diffusion-bonding unit

mosphere. The unorthodox design of the process chamber, radiator, and precision pressing device coupled with the use of a time-sequence control unit have served to appreciably improve the bond quality, rate of output, and the joining operation proper. The overall dimensions of the machine are 1.3 × 1.1 × 1.9 m, and its mass is 900 kg. The bonding force can be adjusted from 20 to 2 000 N, and the maximum temperature of the workpieces is 1 573 K. One cycle is completed in 1.5-2 h.

The type A306-21 machine (Fig. 4-29) is intended for the precision diffusion bonding of components for electron devices and similar products. The bonding force can be adjusted between 20 and 2 000 N. The maximum temperature of the workpieces is 1 473 K. The machine operates at the rate of one or two cycles per hour. The ultimate pressure in the process chamber is 6.7×10^{-3} Pa. The overall dimensions of the machine are 1.6 × 1.36 × 1.94 m, and its mass is 2 000 kg.

Solar-energy machines. This class of machines is exemplified by the type USP-1.5-1.5 universal solar furnace. It has two mirror concentrators, *2* and *3*, served in turn by one heliostat, *1* (Fig. 4-30). The mirrors are each 1.5 m in diameter, with a focal distance of 0.65 m and a beam angle of 60°. Ideally, the focal spot is 0.016 m in diameter; actually it is 0.018 m. The maximum energy density is 1.5×10^7 W m^{-2}, and the total power of the furnace is 1 kW. The furnace can produce a local temperature of 3 273 K, but in diffusion bonding the joint area need not be heated to such a high temperature. To bring down the temperature, the energy flux can be varied either by partly shading the mirrors or by placing the workpiece beyond the focal distance.

In a solar furnace, the workpiece is heated locally and from one side only, so for proper heating the work must be rotated. Therefore the best choice of products for diffusion bonding in a solar furnace are tubes or rods which can readily be rotated continuously. In joining some pairs of materials it may be expected that diffusion bonding with concentrated radiant energy can produce far better results than electric heating, as there is no exposure to ambient factors.

Resistance-heating machines. An example is the type SDVU-5K which is intended to make bellows, aneroid capsules and similar products by diffusion bonding in vacuum. The workpieces are heated by the passage of current from a welding

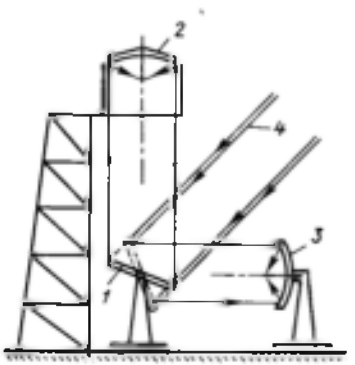


FIG. 4-30 USP-1.5-1.5 universal solar furnace

transformer. In addition to the heating system, the machine includes a vacuum system, a hydraulic system and a control system. The bonding temperature can be set at anywhere between 573 and 1 473 K. The maximum diameter of the weldments is 0.1 m. The machine measures 1.12 × 0.68 × 1.35 m overall [8].

CO_2-shielded resistance diffusion-bonding machines. The use of carbon dioxide as a shielding gas for diffusion bonding has substantially cut down the cost of fabrication for noncritical parts from copper, nickel, lead, medium- and low-carbon steels. This type of machine consists of a process chamber, a scratch-brushing mechanism, a pressing mechanism, a hydraulic mechanism, a bonding current control unit, a CO_2 cylinder manifold, and the associated valves and fittings. All the components are mounted on a common frame. The workpieces are diffusion-bonded in the welding chamber which is hermetically sealed and filled with CO_2. The bonding force is applied to the workpieces by an external hydraulic cylinder and can be varied between broad limits. The scratch-brushing mechanism is placed inside the welding chamber and serves to remove oxide films from the workpieces in the CO_2 atmosphere. The cleaning tool mounted on the arm of the mechanism is introduced between the mating surfaces of the workpieces and is rotated by a d.c. electric motor. The tool can be a scratch brush or a suitably knurled cylindrical milling cutter. CO_2 is fed into the welding chamber from the cylinder via a heater, a pressure regulator and a desiccator. The gas fills the chamber and displaces all air. The machine can butt-join pipes and rods up to 0.05 m in diameter, and also plates 0.05 × 0.015 m in cross section. The maximum total length of the weldments is 0.7 m.

Bibliography

1. AVERIN, I. V., KABANOV, N. N. *Friction Welding in Tool Making.* Mashguiz, Moscow, 1962 (in Russian).
2. BALAKIN, V. I., KHRENOV, K. K. *The Role of Vacuum in Cold Bonding.* "Avtomaticheskaya svarka", 1966, No. 2, p. 79 (in Russian).
3. HEINZE, W. *An Introduction into Vacuum Techniques.* In: Physical Principles of Vacuum Techniques. Gosenergoizdat, 1960, vol. 1 (in Russian).
4. DUSHMAN, Saul. *Scientific Foundations of Vacuum Technique.* 2nd ed. John Wiley & Sons, Inc., New York, 1964.
5. ZAKIROV, F. G., NIKOLAYEV, S. N. *The Exhaust Operator's Reference Book.* Higher School, Moscow, 1973 (in Russian).
6. KAZAKOV, N. F. *Diffusion Bonding in Vacuum.* Mashinostroyeniye, Moscow, 1968 (in Russian).
7. KAZAKOV, N. F., ZHUKOV, V. V. *Equipment for Diffusion Bonding.* PNILDSV, Moscow, 1973 (in Russian).
8. KAZAKOV, N. F. *Diffusion Bonding of Materials.* Mashinostroyeniye, Moscow, 1976 (in Russian).
9. KONYUSHKOV, G. V., KOPYLOV, Yu. N. *Diffusion Bonding in Electronics.* KAZAKOV, N. F. (ed.). Energiya, Moscow, 1974 (in Russian).
10. KOROLEV, B. I., KUZNETSOV, V. I., PIPKO, A. I. *Fundamentals of Vacuum Technique.* Energiya, Moscow, 1975 (in Russian).

11. MINTS, A. L., KRISTI, N. M. *On Some Features of Dynamic Vacuum Systems*. ZhTF, 1956, vol. XXVI, No. 8 (in Russian).
12. CHVERTKO, A. I., NAZARENKO, O. N., SVYATSKY, A. M. *Equipment for Electron Beam Welding*. Naukova Dumka, Kiev, 1973 (in Russian).
13. OLSHANSKY, N. A., SHUBIN, F. V. *Equipment for the Electron-Beam Welding of Metals*. Energiya, Moscow, 1967 (in Russian).
14. PATON, B. Ye. *The Welding of the Future*. In: Science and Mankind. Znaniye, Moscow, 1963, vol. III, pp. 504-513 (in Russian).
15. PIPKO, A. I. *Equipment for the Evacuation of Vacuum Devices*. Energiya, Moscow-Leningrad, 1965 (in Russian).
16. SLUKHOTSKY, A. Ye. *Hardening Inductors*. Mashguiz, Moscow, 1957 (in Russian).
17. SHATOV, A. N., LUKIN, I. V., IVANOV, V. I. *High-Frequency Welding of Metals*. Mashinostroyeniye, Leningrad, 1977 (in Russian).

5

Production Rate. Mechanization and Automation of Equipment

N. F. Kazakov

5.1 Production Rate

One of the ways to raise the production rate of diffusion-bonding equipment is to cut down the time taken up by the various steps in a welding cycle.

The total time per cycle is

$$t_{tot} = t_1 + t_2 + t_3 + t_4 + t_5 + t_6 + t_7 \qquad (5.1)$$

where t_1 is the time to load and mount the work, t_2 is the pump-down time, t_3 is the time to transfer the pressing load to the pieces being joined, t_4 is the heating time, t_5 is the cooling time, t_6 is the time to remove the pressing load, and t_7 is the time to discharge the weldment from the process chamber.

The total time can be cut down either (1) by cutting down the time of each step or (2) by combining or overlapping some or all of the steps. An example of the latter approach is combining t_3 with t_4, and t_5 with t_6: the pressing load is applied to and removed from the workpieces concurrently with their heating and cooling, respectively. In a single-chamber machine the other steps can hardly be combined or overlapped, as this would upset the normal course of the joining operation and impair the quality of the bond. The time to load and discharge a weldment, t_1 and t_7,

11. MINTS, A. L., KRISTI, N. M. *On Some Features of Dynamic Vacuum Systems*. ZhTF, 1956, vol. XXVI, No. 8 (in Russian).
12. CHVERTKO, A. I., NAZARENKO, O. N., SVYATSKY, A. M. *Equipment for Electron Beam Welding*. Naukova Dumka, Kiev, 1973 (in Russian).
13. OLSHANSKY, N. A., SHUBIN, F. V. *Equipment for the Electron-Beam Welding of Metals*. Energiya, Moscow, 1967 (in Russian).
14. PATON, B. Ye. *The Welding of the Future*. In: Science and Mankind. Znaniye, Moscow, 1963, vol. III, pp. 504-513 (in Russian).
15. PIPKO, A. I. *Equipment for the Evacuation of Vacuum Devices*. Energiya, Moscow-Leningrad, 1965 (in Russian).
16. SLUKHOTSKY, A. Ye. *Hardening Inductors*. Mashguiz, Moscow, 1957 (in Russian).
17. SHATOV, A. N., LUKIN, I. V., IVANOV, V. I. *High-Frequency Welding of Metals*. Mashinostroyeniye, Leningrad, 1977 (in Russian).

5

Production Rate. Mechanization and Automation of Equipment

N. F. Kazakov

5.1 Production Rate

One of the ways to raise the production rate of diffusion-bonding equipment is to cut down the time taken up by the various steps in a welding cycle.

The total time per cycle is

$$t_{tot} = t_1 + t_2 + t_3 + t_4 + t_5 + t_6 + t_7 \qquad (5.1)$$

where t_1 is the time to load and mount the work, t_2 is the pump-down time, t_3 is the time to transfer the pressing load to the pieces being joined, t_4 is the heating time, t_5 is the cooling time, t_6 is the time to remove the pressing load, and t_7 is the time to discharge the weldment from the process chamber.

The total time can be cut down either (1) by cutting down the time of each step or (2) by combining or overlapping some or all of the steps. An example of the latter approach is combining t_3 with t_4, and t_5 with t_6: the pressing load is applied to and removed from the workpieces concurrently with their heating and cooling, respectively. In a single-chamber machine the other steps can hardly be combined or overlapped, as this would upset the normal course of the joining operation and impair the quality of the bond. The time to load and discharge a weldment, t_1 and t_7,

can be cut down only slightly, so they are assumed to be constant for a given type of machine. The greater proportion of the total time is taken up by t_2, t_4 and t_5, so, in designing a machine or the joining operation, everything should be done to cut down these items of the time balance.

In the general case, the production rate, P (pieces per min), for diffusion-bonding machines is defined as

$$P = 60Rmn/t_{tot} \tag{5-2}$$

where R is the number of positions, or stations, within a process chamber, m is the number of chambers on a machine, and n is the number of workpieces being joined at the same station. As is seen, the production rate increases with increasing values of R, m and n, and with decreasing t_{tot}. It is clear, therefore, that the best way to raise production rate is to use multiple chambers, multistation jigs and fuxtures, and packeting of workpieces.

The time to load and discharge workpieces accounts for about 4% to 8% of the total cycle time, and it can hardly be reduced by an appreciable amount either by updating the existing machines or by designing new ones. Yet there are ways and means which ought not to be ignored. A good deal can be done by properly designing the layout and size of access ports and covers, machine frames, chamber lids, etc. Before the final engineering step, each design must be tried out on a mock-up. A sizeable proportion of time is lost in setting up and aligning workpieces relative to the heat source, and in connecting thermocouples.

5.2 Jigs and Fixtures

An attractive way out is to use fit-up jigs whose efficiency increases with increasing number of stations. Proper alignment of the work relative to the heat source can be facilitated through the use of movable stages located inside the process chamber(s).

Figure 5-1 shows a fixture to clamp and position the workpieces relative to the heat source, *5* (an inductor). It consists of an upper power cylinder *1*, intermediate rods *2* and *8* joined by a ball-and-socket *3*, a water-cooled stage *7*, a vacuum chamber *4*, and a lower power cylinder *9*. The pieces to be joined, *6*, are set up on the water-cooled stage *7* inside the vacuum chamber. The stage is coupled to the rod *8* of the lower power cylinder. When oil is introduced into the lower space of the lower cylinder, its rod bears against the stage and lifts it until the workpieces take up the desired position relative to the heat source. When the pieces have been positioned and the chamber exhausted, oil is introduced into the upper power cylinder, and its rod acts on the intermediate rod *2* and the ball-and-socket to bring pressure to bear upon the workpieces. The ball-and-socket makes up for the likely inaccuracy in the geometry of the vacuum chamber and ensures that the pressure is evenly distributed over the entire joint area. If, during the heating step, the operator sees that the bond area is not properly oriented relative to the heat source or if one of the pieces must be heated more than the other, he can admit oil to the upper or lower power cylinder in order to move the intermediate rods and the water-cooled stage as

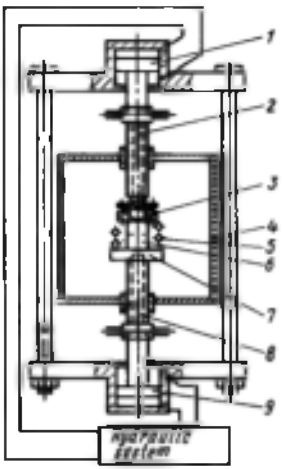


FIG. 5-1 Clamping fixture using counterpressure cylinders

required. The fixture substantialy reduces the time taken up to position the work relative to the heat source and makes it possible to use smaller power cylinders.

As often as not, workpieces of unusual shape or size can be diffusion-bonded with difficulty, if at all, unless some sort of jig or fixture is used. Sometimes, the function of a jig is solely to hold the work or its components in a desired position. In other cases, it may be used to make up for lack of proper contact between the mating surfaces. Whatever the function of a jig, however, its ultimate goal is to raise the rate of fabrication by bonding.

As a rule, jigs are made of high-temperature steels and alloys, ceramic materials, and refractory metals (such as tungsten or molybdenum) — if they were made of ordinary structural materials, they would readily deform and lose shape. Another requirement is that the material of a jig should not seize to the workpiece. This can be avoided by placing an interlayer (such as a parting wash) or mica spacers between the jig and the work. Most frequently, a good parting wash can be prepared from a solution of chalk or a hydrophobic silicone fluid which is then smeared as a thin coat to the mating surface. At 573 K the fluid turns into a refractory film with a fusion point of over 1 273 K; it will effectively prevent diffusion where it is not desired. Unfortunately, parting washes pose another problem — that of polluting the space within the process chamber(s) and impairing the vacuum. To circumvent the problem of pollution, seizure is sometimes avoided by oxidizing the hot components of the jig in air or wet hydrogen. The oxide film is, however, less effective than a parting wash.

At present, a great variety of jigs have been designed for use in diffusion bonding. Figure 5-2 shows one intended for the joining of thermocouples; the jig both raises the production rate and makes up for inaccuracies in the workmanship. This type of jig has six positions (stations), so as many as six thermocouples can be bonded in a single cycle with it (only one station is shown). The thermocouple ends are

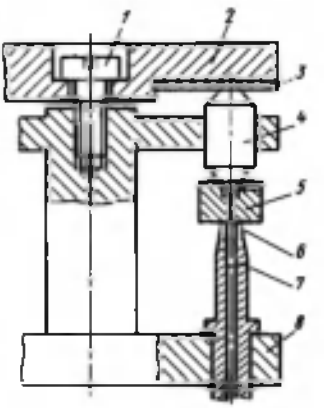


FIG. 5-2 Thermocouple bonding fixture
1 — screw; *2* — pressure ring; *3* — spacer; *4* — compression rod; *5* — hold-down; *6*, *7* — thermocouple components; *8* — baseplate

secured in molybdenum clamps preheated in air in order to produce a stable oxide film preventing seizure between the clamp and the thermocouple material. The clamps bear against rods which support a copper spacer. On heating, the copper softens and compensates for lack of ideal contact between the rods and the spacer. In this way, pressing load is uniformly transferred to the parts being joined. The copper spacer is topped by a pressure ring which is held down by a screw so that the jig and all the thermocouples prepared for bonding are firmly held together as a unit. The assembly is then placed in an inductor and heated to the desired temperature. The pressure ring, screw, rods, and baseplate of the jig are made of a corrosion-resistant steel.

A difficulty arises in cases where one of the pieces being joined is a foil. It is hardly possible to produce an even pressure on the foil unless a suitable jig is used. Another difficulty is to maintain a uniform temperature field within the thin member. A jig designed for the purpose is shown in Fig. 5-3. It consists essentially

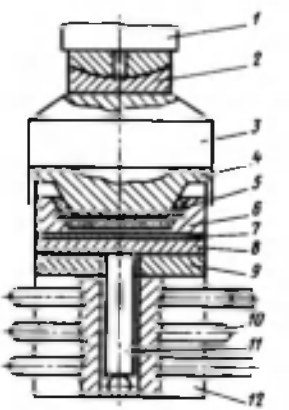


FIG. 5-3 Foil bonding fixture
1 — compression rod; *2* — support; *3* — upper half of die; *4* — guide; *5* — sand; *6* — lower half of die; *7* — foil; *8* — workpiece; *9* — washer; *10* — inductor; *11* — core; *12* — baseplate

of a baseplate and a split die. An inductor is wound around the base in such a manner that a small iron-cored induction-heating furnace is formed. The foil is heated by thermal conduction from the iron core through the other piece of the weldment over the entire contact area. The upper part of the split die is linked by a ball-and-socket to the rod of a hydraulic press. The lower part of the die is a flexible plate with stiffened edges. With this jig, platinum foil 0.01-0.05 mm thick can readily be diffusion-bonded to a titanium piece over an area of over 100 cm^2.

Special problems arise in diffusion-bonding bimetallic tubes. In such applications, jigs utilize the difference in coefficients of thermal expansion.

5.3 Reduction of Pump-Down Time

All other conditions being equal, the pump-down time depends on the ultimate pressure to be achieved. In diffusion-bonding machines, the pump-down time accounts for 15-50% of the total cycle time t_{tot}. At an ultimate pressure of $6.5 \times \times 10^{-5}$-$1.3 \times 10^{-6}$ Pa the pump-down time may take up as much as 80% to 90% of t_{tot}. A straightforward reduction of pump-down time would raise the ultimate pressure, and this is ruled out by the very nature of the diffusion-bonding process. A way out is to improve the design and workmanship of the vacuum system as a whole and of its components, and to operate the vacuum system in an optimal manner. Among other things, it is important to pick the right size of pumps, to use the right kind of materials for seals and gaskets (especially for those facing the vacuum), and to reduce the volume to be exhausted. The capacity of the exhaust facilities can be determined by calculation or by experiment, and it ought not to be specified too high, as this would add to the cost and the size of the diffusion-bonding machine. Special emphasis should be placed on the conductance of the vacuum pipework as it strongly affects the pumping speed of the system. Apart from the proper choice of pumps and materials, an important reserve of cutting down the pump-down time is to reduce the evacuated volume. Sometimes it would pay to use vacuum locally, that is, just around the joint area. Such local chambers are small in size and their pump-down time is negligible. The bonding zone in such chambers is sealed with suitable gasketing materials or putties.

5.4 Continuity of Pressing Load Transfer

The bonding force is transferred to the workpieces by the pressing system in a precisely specified direction. Once fitted up for bonding, the workpieces may not be moved at right angles to the direction of the pressing load. Therefore, the machine can only operate periodically — no other piece can be bonded in the same station until the previous one has been joined. The situation is different when the workpieces, while held together by the bonding force, are allowed to move relative to the heat source — then the machine will operate continuously, and a higher production rate will thus be assured.

An arrangement with which a diffusion-bonding machine can operate continuously is shown in Fig. 5-4. The workpieces *1* are mounted on a spacer of a

plastic metal *4*, which fits to a stiff auxiliary plate *3* made of a high-temperature alloy. From above the workpieces are covered by another auxiliary plate, *3*. The two auxiliary plates are held together. The assembly is then threaded between rolls *2*, and the rolls advance the assembly. Placed parallel to the auxiliary plates are radiators *5* (which may be molybdenum or tungsten rods, infrared lamps, etc.). While slowly moving between the rolls, the workpieces travel past the heat sources, rise to the bonding temperature, and are maintained at that temperature for the specified span of time. At the end of the heating time, the workpieces are allowed to cool to the specified temperature between the rotating rolls. The spacing between the rolls depends on the stiffness of the auxiliary cover plates. By varying the number of rolls and the spacing between them, it is possible to vary the joining speed at will. To avoid the excessive heating of the cover plates, cooling may be applied to the rolls.

Figure 5-5 shows another fixture which serves to make diffusion bonding a continuous process. It is intended to fabricate multilayer bands of large length from various metals and alloys. The bands to be bonded together, *1* and *2*, are wound on reels fitted with suitable tensioning devices. When the take-up drum, *6*, is turned on, the bands start moving and are heated from a heat source *3*. The tensioning force applied to band *1* (p_1) is smaller than that applied to band *2* (p_2). Therefore, on moving round roller *4* band *2* exerts on band *1* a force sufficient for a diffusion bond to be made. In travelling through the arc of wrap, the two bands move at the same linear velocity, so they remain stationary relative to each other, but are moving relative to any other point in the vacuum chamber. The velocity of roller *4* and drum *6* is determined by the ratio of the arc of wrap to the time it takes a diffusion bond to form between the bands. The bonded band is cooled at roller *5* and wound on the take-up drum. If the band is not thick enough to exert a sufficient bonding force, the latter can be supplied by a strong auxiliary band *7*.

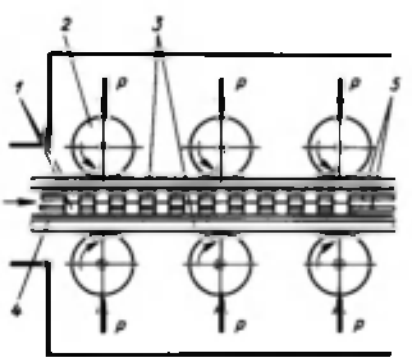


FIG. 5-4 Arrangement for continuous bonding

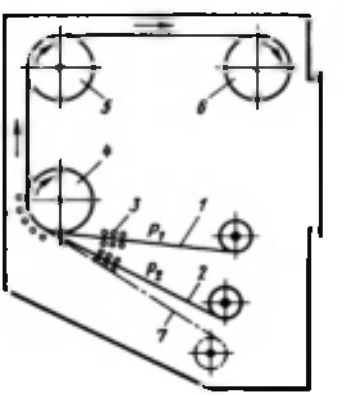


FIG. 5-5 Arrangement for continuous bonding of bimetallic band

5.5 Reduction of Heating and Cooling Time

For a diffusion bond to form, the workpieces should be held at a certain temperature and under a certain bonding force (pressing load). As already noted, heat for diffusion bonding can be supplied by any known sources (induction, radiation, light-beam, electric-arc, etc.). Most often, use is made of induction and radiation heating. Inductors are usually supplied with current by high-frequency tube oscillators operating in the frequency range 60-500 kHz. However, only small workpieces can be heated inductively in this frequency range. For larger pieces, it is customary to use rotary generators supplying current at a frequency of 500-10 000 Hz. Their advantage is that they appreciably reduce the heating time.

Any diffusion-bonding machine is equipped with a cooling system. The desired rate of cooling (or cooling time) is maintained by using water cooling for the supports, vacuum chamber(s), and intermediate rods. Also, the bonding chamber is purged for cooling with an inert gas.

5.6 Combining or Overlapping the Steps

As has been noted, the total time can be cut down by combining or overlapping one step with some other. For example, the evacuation of the process chamber can readily be combined with the heating and cooling of the work. The economic benefit is obvious as the production rate is increased several-fold. An example is the fixture developed by this author and schematically shown in Fig. 5-6. It consists of a housing *1*, two power cylinders, *2* and *10*, intermediate rods, *3* and *9*, each complete with seals, an inductor *4*, a faceplate *6*, a vacuum chamber *7*, and a shaft *8*. The faceplate set up inside the vacuum chamber is divided into several stations each of which receives the parts to be joined, *5*. At the loading, joining and discharge steps, the shaft and the faceplate are turned through the desired angle. The vacuum

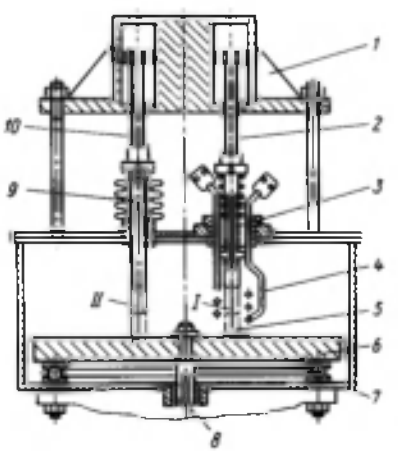


FIG. 5-6 Bonding fixture with a rotating faceplate

chamber has two stations, one for welding (*I*) and other for cooling (*II*). As the faceplate is turned, the parts being joined are positioned into station *I*, whereas the inductor and intermediate rod *3* are raised to the top position. When oil is introduced into power cylinder *2*, its rod goes down, and so do the inductor and intermediate rod *3*. The parts being joined are heated inside the inductor and clamped together by rod *3*. After the bond is completed, the weldment is moved into station *II* where it is allowed to cool under the load applied by power cylinder *10* whereas a new workpiece is installed in station *I*. In this way, the cycle is repeated until all the parts set up on the faceplate are bonded. The fixture substantially raises the production rate of single-chamber machines owing to a cut in the pump-down time and by overlapping the heating and cooling steps.

Sometimes, it is advantageous to make diffusion bonds in several chambers rather than in one. As an example, Fig. 5-7 shows a rotary-type five-station machine in which the operating cycle is divided into five steps, namely loading-discharge, roughing-down, evacuation to a high vacuum, bonding (diffusion treatment), and cooling. The rotary table is actuated by a drive consisting of an electric motor *11* and a variable-speed reduction gear *12*. The main shaft of the machine *1* is free to pass through a stationary disc *2* which gives support to a rotary disc *3* made fast to the main shaft water-cooled and sealed by a rotating stuffing box *10*. The two discs are carefully lapped to each other and are each divided into several stations or positions. The five stations of the rotary disc carry vacuum chambers, *5*, connected by vacuum lines *4* to openings in the disc. Heat for diffusion bonding is supplied by the inductors *7*, and the pressing load is supplied by a hydraulic pump *8* via hydraulic cylinders *6*. The machine operates as follows. When the machine is in the "load-discharge" position, the parts to be joined are installed in a vacuum chamber. After

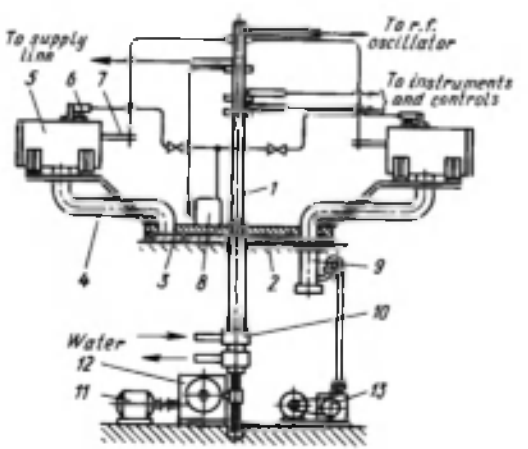


FIG. 5-7 Five-station rotary-type diffusion-bonding unit

the chamber has been properly sealed, the rotary disc transfers it to the "rough-down" position. In that position, the chamber is exhausted to a vacuum of around 6.5 Pa by a forevacuum pump *13* and is then moved to the "high-vacuum" position where it is exhausted by an oil-vapour pump *9* to an ultimate pressure of $1.3 \times \times 10^{-1}$-$1.3 \times 10^{-2}$ Pa. In the "weld" position, the inductor is connected to an r.f. oscillator, and the hydraulic cylinder to the oil hydraulic pump. The parts being joined are heated and pressed together as required. From the "bond" position the vacuum chamber is moved to the "cool" position where the weldments are allowed to cool to 373-473 K. Then again comes the "load-discharge" position where the diffusion-bonded parts are removed and those to be joined installed in the chamber. In this way, the various steps of the cycle occur all at the same time, but in different positions. As a result, the machine operates at a very high rate.

Figure 5-8 shows a simplified diagram of an airlock-type diffusion-bonding machine. In it, the pressing load is developed by thermally induced interference. The parts to be joined are installed in holders *4* made from a material having a smaller coefficient of linear expansion than the workpieces. The loaded holders are placed into a chamber *I* which is exhausted to a vacuum of 1.3×10^{-1} Pa. The same vacuum is produced in another chamber, *IV*. At the same time, chambers *II* and *III* are evacuated to the specified pressures. After the desired vacuum has been reached in chamber *I*, a gate *3* is opened and a pusher *1* moves the workpieces into chamber *II*. Since chamber *I* has been exhausted to about the same vacuum as chamber *II*, the transfer of the work affects the pressure in the latter very little, if at all. In chamber *II*, the parts in their holders are heated from a source *6*, and the required bonding pressure is produced by the difference in coefficient of linear expansion between the holders and the work. After the bond is completed, another gate *5* is opened, and a second pusher *2* transfers the weldments into chamber *III* where they are allowed to cool to 373-473 K. After cooling, the weldments are collected in chamber *IV* until the required number has been accumulated and is discharged. During the discharge and load step chambers *I* and *IV* are open to the atmosphere, whereas chambers *II* and *III* remain under vacuum all the time.

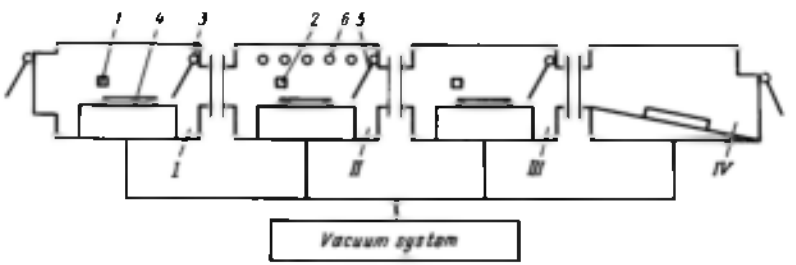


FIG. 5-8 Airlock-type diffusion-bonding unit

5.7 Automatic Control of Bonding Conditions

As an example, consider the automatic control system shown in Fig. 5-9. A light annunciator displays all the relevant process variables which are controlled in an interrelated manner and recorded on a paper chart. These variables are the ultimate pressure (vacuum), the pressing load, the temperature and duration of the diffusion treatment, the cooling temperature, and the cooling time of the machine at the end of a shift. The bonding temperature is measured directly in the bonding zone either by a thermocouple or by a photopyrometer.

The above system is embodied in the type SDVU-12 machine which operates automatically except during the start-up at the beginning of a shift when the operator turns on cooling water. Water feed is monitored by a pressure gauge and controlled by a pressure relay. The pressure relay will not permit the operator to start the machine unless cooling water has been turned on and its pressure is as it should be. This relay also starts all the units of the machine. Pressure on the "Start" button initiates an automatic cycle by closing the circuit to a bank of pilot relays *1* and a magnetic starter *2* which in turn energizes the electric motor *3* of a roughing-down vacuum pump. At the same time, a vacuum valve *4* opens, and the "Roughing-down line open" sign illuminates on the light annunciator *16*. The first stage of an r.f. generator *5* (the heat source) is energized and current is fed to a time relay installed inside the generator, which turns on the second stage of the generator with a time delay of 30 s and allows it to warm up. If any other source is used instead of an r.f. generator, no warm-up time will be required, and it may be turned on directly to heat the parts being joined. The vacuum in the process chamber is measured by a vacuum sensor *6* which transmits the transduced vacuum signal to a multipoint vacuum indicator-recorder-controller *7*. When the vacuum in the process chamber *8* has reached 6.5 Pa, the controller generates a signal which energizes an auxiliary relay *9* and pilot relays *1* which apply power via an autotransformer *10* to the electric heater *11* of an oil-vapour pump. Also, the pilot relays act via a reversible starter *12* to open flap valve *13* and vacuum valve *14*, and the "Roughing-down line open" sign on the annunciator goes out. When the vacuum chamber has reached a vacuum of 1.3×10^{-2} Pa, the vacuum sensor transmits an appropriate signal to the controller which energizes the auxiliary relay *9*, and this causes operation of pilot relays *1* which turn on the r.f. generator (the heat source) to heat the workpieces *15*. As this happens, the "Heat on" sign illuminates on the annunciator.

Should the vacuum in the process chamber become less than 1.3×10^{-1} Pa, the controller will turn off the heat source, and will turn it on again after the specified vacuum has been restored. When the r.f. generator is turned off, the "Heat on" sign on the annunciator goes out. The temperature of the workpieces is monitored, recorded and controlled by a controller *17* connected to the workpieces by a thermocouple *18* (or a photopyrometer). When the workpieces have reached the specified bonding temperature, controller *17* feeds a signal via auxiliary relay *9* and pilot relays *1* to a magnetic starter *29* which turns on the electric motor *19* of the hydraulic pump, whereas solenoid *20* moves the spool valve so that it feeds oil to the top of the hydraulic cylinder *21* which exerts the requisite bonding pressure to the

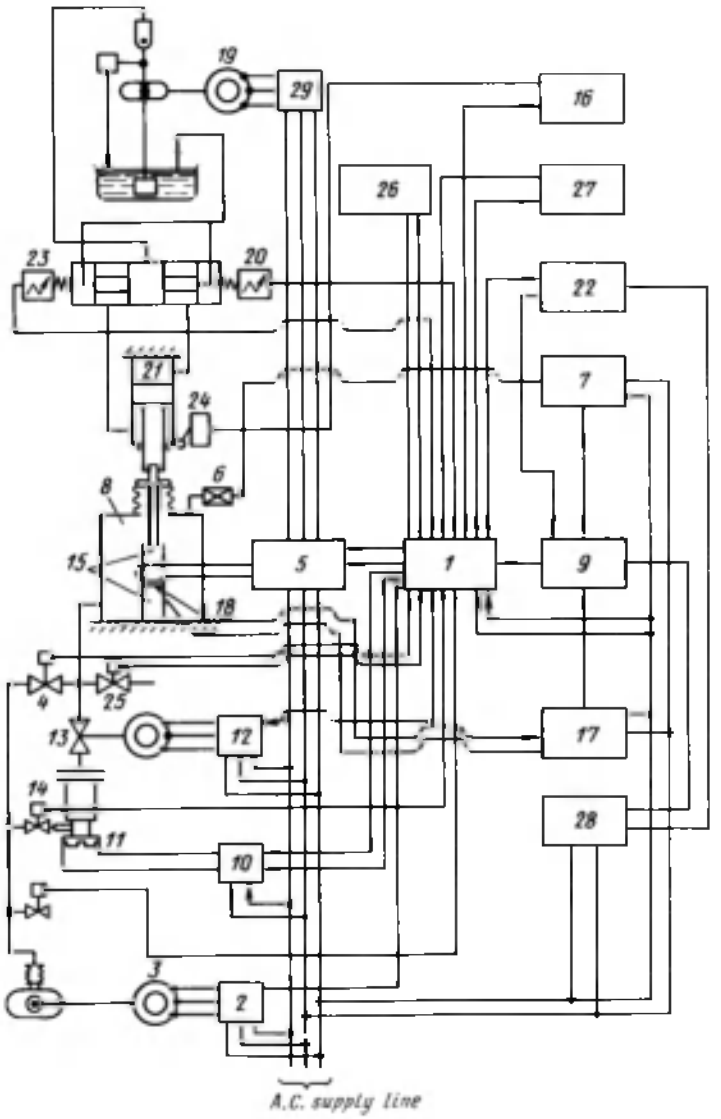


FIG 5-9 Diffusion bonding automatic control system

workpieces. Also, a timer *22* is energized to count the bonding time. After the specified time passes, the timer applies a signal to auxiliary relay and pilot relay *1* to turn off the heat source, the "Heat on" sign on the annunciator goes out, and the weldment is allowed to cool down to the specified temperature (or for a predetermined time). When the weldment has cooled to the required temperature, controller *17* sends a signal via auxiliary relay *9* and pilot relays *1* to solenoid *23* which moves the spool valve so as to lift the compression rod, and the pressing load is removed from the weldment. At the same time, a limit switch *24* illuminates the "Rod up" sign on the light annunciator. In addition to removal of the pressing load from the weldment, the pilot relays energize the reversible magnetic starter *12* to close the flap valve *13* thereby turning off the "High vacuum open" sign on the hight annunciator; control the autotransformer so that the voltage impressed on the electric heater *11* is halved; open valve *25* to admit air in the vacuum chamber, and start the timer *26* to count the air admission time. At the end of the air admission interval, the timer *26* acts via pilot relays *1* to de-energize solenoids *20* and *23* and causes magnetic starter *29* to stop the electric motor *19* of the hydraulic pump. At the end of the shift, the machine can be stopped, by pressing the "Stop" button. This will cause pilot relays *1* to turn off the electric heater *11* of the oil-vapour pump and to start the timer *27* of the machine. After a delay of 30 min, the timer *27* will de-energize all of the machine, including the r.f. generator. Power for auxiliary relay *9* and heating timer *22* is supplied by a power unit *28*.

6

Diffusion Bonding of Steels

N. F. Kazakov

6.1 Bonding of Similar Steels

Low-carbon steels lend themselves readily to welding. Nevertheless, special care must be taken in order to make a full-strength joint and to avoid defects in the weld metal. The mechanical properties of the joint depend on its structure, and this is in turn decided by the chemical composition of the material, welding conditions, and postweld heat treatment, if any is given.

Welding joints made in low-carbon steels by any fusion welding processes show a satisfactory resistance towards solidification cracking which is attributed to the low carbon content of the weld metal. As the carbon content is increased, however, the weldability is impaired. With over 0.3% C, steels are susceptible to superheating, chilling, cold cracking in the joint, and porosity in the weld metal.

workpieces. Also, a timer *22* is energized to count the bonding time. After the specified time passes, the timer applies a signal to auxiliary relay and pilot relay *1* to turn off the heat source, the "Heat on" sign on the annunciator goes out, and the weldment is allowed to cool down to the specified temperature (or for a predetermined time). When the weldment has cooled to the required temperature, controller *17* sends a signal via auxiliary relay *9* and pilot relays *1* to solenoid *23* which moves the spool valve so as to lift the compression rod, and the pressing load is removed from the weldment. At the same time, a limit switch *24* illuminates the "Rod up" sign on the light annunciator. In addition to removal of the pressing load from the weldment, the pilot relays energize the reversible magnetic starter *12* to close the flap valve *13* thereby turning off the "High vacuum open" sign on the hight annunciator; control the autotransformer so that the voltage impressed on the electric heater *11* is halved; open valve *25* to admit air in the vacuum chamber, and start the timer *26* to count the air admission time. At the end of the air admission interval, the timer *26* acts via pilot relays *1* to de-energize solenoids *20* and *23* and causes magnetic starter *29* to stop the electric motor *19* of the hydraulic pump. At the end of the shift, the machine can be stopped, by pressing the "Stop" button. This will cause pilot relays *1* to turn off the electric heater *11* of the oil-vapour pump and to start the timer *27* of the machine. After a delay of 30 min, the timer *27* will de-energize all of the machine, including the r.f. generator. Power for auxiliary relay *9* and heating timer *22* is supplied by a power unit *28*.

6

Diffusion Bonding of Steels

N. F. Kazakov

6.1 Bonding of Similar Steels

Low-carbon steels lend themselves readily to welding. Nevertheless, special care must be taken in order to make a full-strength joint and to avoid defects in the weld metal. The mechanical properties of the joint depend on its structure, and this is in turn decided by the chemical composition of the material, welding conditions, and postweld heat treatment, if any is given.

Welding joints made in low-carbon steels by any fusion welding processes show a satisfactory resistance towards solidification cracking which is attributed to the low carbon content of the weld metal. As the carbon content is increased, however, the weldability is impaired. With over 0.3% C, steels are susceptible to superheating, chilling, cold cracking in the joint, and porosity in the weld metal.

Medium-alloy steels are good for noncritical parts. After welding, such parts are sometimes quenched and tempered so as to obtain an ultimate tensile strength of 686-882 MPa.

As to structural steels, there is no generally accepted criterion of their weldability. The usual considerations in their choice are the mechanical properties of the parent and weld metal, ease of pressing, resistance towards hot and cold cracking in the course of welding, hot and cold workability, labour requirements, etc. The top requirement, however, is the reliability of welded joints.

Carbon and low-alloy steels should preferably be welded by processes that assure high strength, retain the original properties in the metals being joined, and avoid the defects usually associated with fusion welding processes. This is above all true of vacuum diffusion bonding which is carried out at temperatures well below the fusion point. In the circumstances, primary crystallization is nonexistent, and the danger of hot cracking is mitigated. Since, in vacuum diffusion bonding, the oxide films on the mating surfaces are caused to sublime or diffuse into the bulk of the parent metal, the remaining oxide film has practically no effect on the properties of the joint.

An example is steel-20. Ordinarily, it is easy to weld by any process. When made into spinning rings, however, several difficulties arise. Sometimes, spinning rings are fabricated by turning from rods, from seamless tubing, or by powder metallurgy. With rods, as much as 80 to 90% of the stock is wasted as chips; with powder metallurgy, the waste accounts for 26-30%. With welded rings, there is practically no waste, but one has to meet a number of stringent requirements. Among other things, there must be a precise axial and radial alignment at the joint, the joint itself should not be practically visible, there should be no deformation, burning or other welding defects, the weld metal must have a high strength and the same wear resistance as the parent metal. These requirements are best of all satisfied by diffusion bonding in vacuum. As has been found, the optimal conditions are $T = 1\,223$ K; $p_b = 15.7$ MPa, $t = 6$ min, and $p_{vac} = 1.3 \times 10^{-2}$ Pa. Diffusion-bonded rings show no visible interface at the joint which is an indication that diffusion has been complete. Visual inspection reveals no axial or radial misalignment, excessive deformation, etc.

An extensive study into the diffusion bonding of alloyed structural steels, such as 12X2H4A, 18X2H4BA, 30XГCA, 30XГCHA, 30X2H2M and 18X2M steel, has been carried out at the Paton Electric Welding Institute (Kiev, USSR). When welded under optimal conditions ($T = 1423$-1453 K, $p_b = 9.8$ MPa, $t = 12$ min, and $p_{vac} = 1.3 \times 10^{-3}$ Pa), full-strength joints have been obtained in 12X2H4A and 30XГCA steels. The impact toughness is sometimes lower than it is for the parent metal. Overheating might result in embrittlement of the bond, but the condition can be rectified by postheat treatment. The impact toughness has been found to depend on the bonding force applied, being a maximum at an optimal bonding force which corresponds to a maximum value of the diffusion coefficient.

With regard to tool steels, the primary requirement is the utmost in the hardness of cutting tools. Most often, tools are made from high-speed tool steels. When

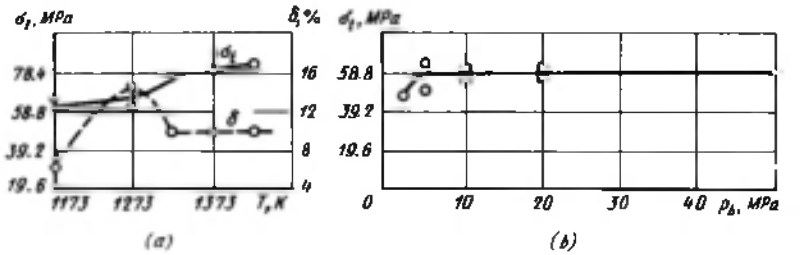


FIG 6-1 Effect of (*a*) bonding temperature and (*b*) pressing load on the strength of joints in P18 steel

fabricated by fusion and resistance welding, the tools display a sweated surface and a ledeburite structure at the interface. Also, the weld is decarburized, and impoverished in carbides and alloying elements. In contrast, the diffusion bonding of P18 high-speed tool steel under optimal conditions (T = 1 373 K, p_b = 9.8 MPa, t = 5 min, and p_{vac} = 0.13 Pa) produces quality joints (Figs. 6-1 and 6-2). Inspection reveals no sweated surfaces or a ledeburite structure even in the very thin layers at the interface. No physical interface between the mating parts is found. Postheat treatment (annealing) more than doubles the strength of the joint (Fig. 6-3). This procedure is employed to reclaim cutting tools fabricated from P18 steel.

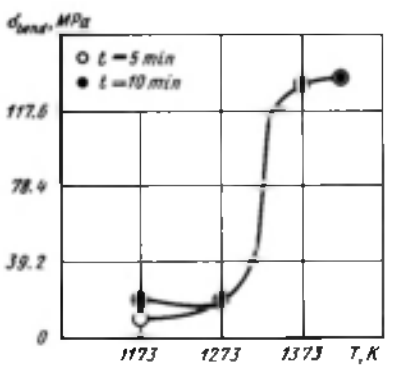


FIG 6-2 Bending strength of joints in P18 steel as a function of bonding temperature; p_b = 20 MPa

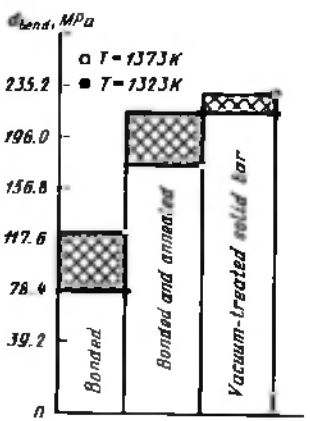


FIG. 6-3 Bending strength of joints made at p_b = 10 MPa and t = 5 min. The shaded areas give spread in experimental data

Of high-alloy steels, those most readily weldable and widely used in weldments are austenitic steels, such as 18% Cr-8% Ni austenitic steels. In the above formulation, these steels are used as corrosion-resistant. With higher percentage of Cr and Ni they are turned into high-temperature and heat-resistant steels. The carbon content of Cr-Ni steels must be not over 0.1-0.15%, since otherwise chromium carbides can precipitate, thereby impairing the valuable qualities of austenitic steels.

The welding of an austenitic steel must as a rule be conducted so as to retain its austenitic structure in the joint and all the associated properties, such as high corrosion resistance, high ductility, etc. The decomposition of the austenite is promoted by heating the metal to over the austenitic transformation point, by a reduction in the percentage of austenite-forming elements, increased carbon content (in the case of low-alloy austenitic steels), excessive contamination by impurities, etc. This is why, in welding an austenitic steel, it is important to keep the heating time and the heat input to a minimum. Special care must be taken to avoid hot cracks as they result in the failure of the weldment immediately after welding or in service. In principle, austenitic steels can be welded with an electric arc and coated electrodes, by the submerged-arc process, by the electrogas or electroslag process, by plasma and electron beam welding.

Martensitic and martensitic-ferrite steels are readily and deeply hardenable, so they need postheat treatment, usually low or high tempering. Frequently preheating may be necessary. In fusion welding, every effort is made to enhance the ductility of the deposited metal and to avoid cracking. To this end, electrodes or filler rods from austenitic steels are often used in arc welding.

The principal drawback of welds in high-chromium ferrite steels is coarse grain next to and, sometimes, in the weld metal, because of which a good deal of embrittlement occurs.

Because of the difficulties in making quality welds, ferrite steels are frequently replaced with Cr-Ni ferrite-austenitic steels with 40-60% ferrite in their structure.

The vacuum diffusion-bonding of high-alloy steels avoids the drawbacks inevitable with other welding processes. This will be clear from the examples that follow. One is the fabrication of standard leaks used in the testing (leak hunting) and operation of vacuum equipment and of vacuum devices. The flow rate of a fluid through a leak can be maintained at a steady value only if its geometry and surface roughness do not vary with time. Glass and quartz capillary tubes are not suitable for use in an industrial environment. A far better choice is the standard leak made in the shape of a triangular channel on the surface of a cylinder 20 mm in diameter and 10 mm high. The cylinder is fabricated by the diffusion bonding of two halves of austenitic stainless 12X18H10T steel, and the triangular passage is shaped on one of the two halves prior to joining. After the two halves are bonded together, the passage appears as a pore in the metal. In fact, a leak of any size can be thus made, as the joining process does not involve melting at the contact area and the amount of upset can readily be controlled by varying the pressing load. The depth of the passage and its surface roughness depend on the extent of deformation in the course of bonding and the original roughness of the blanks.

A study has been carried out to assess the effect of the depth of the passage made prior to bonding and the diffusion-bonding conditions on the effective radius of the standard leak. The diffusion-bonding conditions used in the study included temperatures of 1 073 K, 1 173 K, 1 273 K, and 1 373 K, pressing loads of 4.9 MPa, 9.8 MPa and 19.6 MPa, holding times of 5 and 10 min, and a vacuum of 2.6×10^{-2}-3.2×10^{-2} Pa. The optimal conditions for 12X18H10T steel appear to be $T = 1\,273$ K, $p_b = 19.6$ MPa, $t = 10$ min, and $p_{vac} = 2.6 \times 10^{-3}$ Pa. The standard leaks joined under the optimal bonding conditions successfully stood all tests.

In another study, diffusion bonding conditions have been assessed for 45X14H14B2M steel. The objective of the study was to follow the effect of the bonding conditions on the quality and performance of the material. Several values of temperature were used, each in the anticipation of the desired rate of plastic deformation, the course of the diffusion processes, resultant structural transformations, grain growth and mechanical properties; this choice of temperature was necessitated by the wide range of alloying elements used. The optimal bonding temperature was found to be higher than for carbon or low-alloy steels. The other

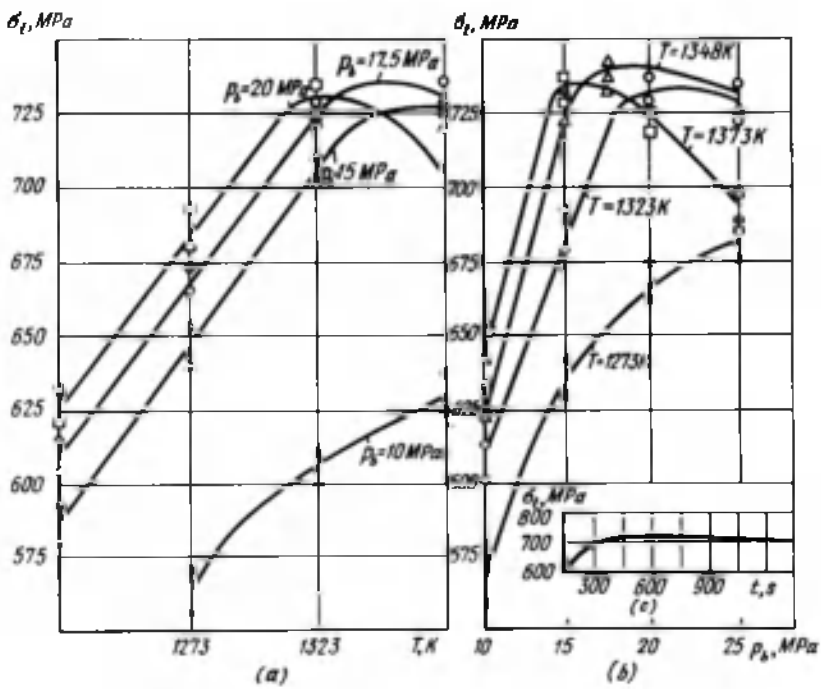


FIG 6-4 Effect of (*a*) bonding temperature, (*b*) pressing load and (*c*) duration of pressure on the ultimate tensile strength, σ_t, of joints in 45X14H14B2M steel. In (*a*) and (*b*), $p_{vac} = 1.33 \times 10^{-2}$ Pa, $t = 10$ min

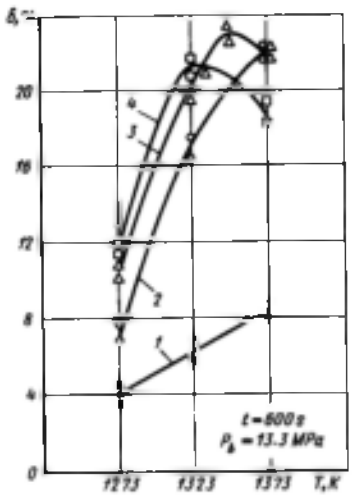


FIG. 6-5 Elongation, δ, %, of joints in 45X14H14B2M steel as a function of bonding temperature ($p_{vac} = 1.33 \times 10^{-2}$ Pa, $t = 10$ min) $1 - p_b = 10$ MPa; $2 - p_b = 15$ MPa; $3 - p_b = 17.5$ MPa; $4 - p_b = 20$ MPa

bonding conditions investigated were pressing load, holding time, and vacuum in the bonding chamber (Fig. 6-4). Strong and sound bonds in 45X14H14B2M steel were obtained under optimal conditions which included $T = 350$ K, $p_b = 1.7 \times 10^7$ Pa, $t = 10$ min, and $p_{vac} = 9.8 \times 10^{-4}$ Pa, with no trace of interface visible. The impact toughness of the joints made under optimal conditions proved not lower than for the parent metal (Fig. 6-5).

On the basis of the above studies it may be concluded that in the course of welding the structure of the metal varies due to intergranular and intragranular creep and diffusion processes. High pressing load substantially increases the rate of creep, accentuates intergranular creep and grain growth in the bond, and all this intensifies recrystallization processes.

To obtain joints having optimal qualities in the diffusion bonding of austenitic steels, it is necessary to promote recrystallization, but to a certain limit only. This limit is set by grain growth and the redistribution of alloying elements. Both impair the impact toughness of the joint.

The above procedure has been used to make vacuum valves from 45X14H14B2M steel [1, 2]. The products were tested for mechanical properties under conditions close to those existing in actual service, above all in the temperature range 1023-1073 K, with special emphasis on the ductility of the bond and the factors affecting it. As tests for short- and long-term strength, creep strength, fatigue strength, thermal stability, resistance to gas corrosion, ductility at failure, and vacuum tightness have shown, the bond thus obtained meets all the requirements under service conditions and that it can operate at varying load and at elevated temperature.

Investigations into the weldability of Cr-Ni austenitic steels have also been carried on 14X14H14B2M, X14H14CB2M, 37X12H8Г8МФБ and 40X15H7Г7Ф2МС

steels. The resultant joints have all been sound and strong and suited for their intended applications.

Diffusion bonds in martensitic steels, such as 14X17H2 and 20X13, have been studied for the effect of temperature, bonding force and holding time on their strength. For 14X17H2 steel the optimal diffusion-bonding conditions have been found (Fig. 6-6) to be as follows: $T = 1\,273$ K, $p_b = 14.7$-25.5 MPa, $t = 20$ min and $p_{vac} = 6.5 \times 10^{-2}$ Pa. During the study, failure occurred at the parent metal, but in the course of welding the 14X17H2 steel showed tendency towards hardening. To remove the locked stresses and the inadvertent hardening, the specimens were given low tempering in a furnace at a point below A_{c3} and above A_{c1}. When tested in tension, the annealed specimens showed a reduction in hardness (Rockwell C 15-20) and an improvement in mechanical properties, especially ductility. This seems to stem from the homogenization of chemical properties in the bond area.

Made under optimal conditions and postweld-treated, the specimens were tested for short-term static strength at elevated temperatures (573 K, 773 K, and 878 K). The bonds in 14X17H2 steel showed strength and ductility somewhat exceeding the nominal values.

The specimens diffusion-bonded under optimal conditions from pieces premachined to a surface roughness of 2.5 μm showed a marked deformation after bonding (3-4%), which is inadmissible in the fabrication of, say, fluidics components. The postweld deformation was reduced by mechanically or electrically polishing the mating surfaces prior to bonding. The deformation was found to be 0.2-0.1%.

The fabrication of reliable elbows for hydraulic lines by the argon-arc process in the martensitic 20X13 steel is a formidable task because the steel tends to crack in the welding zone and to pick up hardness when heated to over 1 273 K. Also, the in-

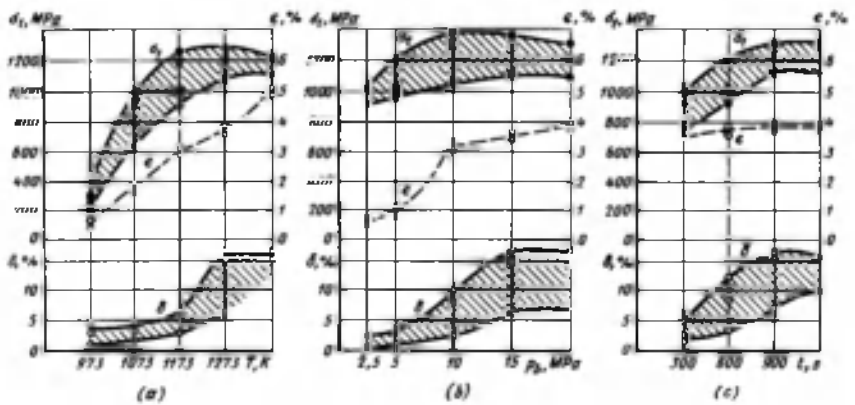


FIG. 6-6 Effect of (*a*) bonding temperature, (*b*) pressing load and (*c*) duration of pressure on the ultimate tensile strength, σ_t, and percent elongation, δ, %, of joints in 14X17H2 steel

ternal stresses produced by structural inhomogeneities lead to haircracking which is out of the question in the elbows since they are subjected to dynamic vibrations under heavy loads in service. No haircracking occurs at a temperature of below 1 273 K. None of the shortcomings listed above exist in diffusion-bonded elbows [1, 2]. When bonded at T = 1213-1233 K, p_b = 15.7 MPa, t = 10 min, and p_{vac} = 0.13 Pa, the elbows have neither haircracks nor any other internal defects. Since, in diffusion bonding, the weldment is vacuum-annealed at the cooling step, no self-hardening occurs in 20X13 steel. When hydrostatically tested under a pressure of 175 MPa, the bonded joints showed neither leaks nor any other defects. When a lot of elbows was tested on a vibration rig at a temperature 288-343 K, a pressure of 31 MPa and a loading rate of 3 cycles per minute, no leaks were found after the test.

6.2 Bonding of Dissimilar Steels

In joining dissimilar steels it is important, in addition to the general weldability considerations, to take into account additional factors affecting the serviceability of welded joints. These factors are the changes brought about by fusion welding in the chemical analysis of the metal adjacent to the parent metal; the appearance of weak and brittle solidification and diffusion interlayers widely varying in composition; and the locked stresses that cannot be relieved when joining structurally different metals. Because of this, a welded joint develops chemical, structural and mechanical inhomogeneities.

In joining dissimilar steels falling in the same structural class, the tempering of a weldment will relieve the welding stresses in much the same way as it does in joining steels having the same range of alloying elements. With steels falling in different structural classes, tempering will inevitably lead to a new field of locked stresses due to the difference in thermal expansion. Taking an austenitic steel joined to a pearlitic steel as an example, cooling after tempering will produce tensile stresses in the austenitic part and balancing compressive stresses in the pearlitic part. The situation is reversed when a pearlitic steel is joined to a high-chromium steel.

Diffusion bonding prevents or markedly reduces mechanical, structural and chemical inhomogeneities in joining dissimilar steels, and also the magnitude and effect of locked stresses.

The potentialities of diffusion bonding of 12X18H9T austenitic steel to 12X13 (martensitic) steel have been investigated in the fabrication of a casing [1, 2]. The welding variables and procedure were tried out on specimens 15 mm in diameter. The optimal conditions were found to be as follows: T = 1 323 K, p_b = 14.7 MPa, t = 20 min and $p_{vac} = 0.13 \times 10^{-2}$-$6.5 \times 10^{-2}$ Pa. The ultimate tensile strength was found to be 627.2 MPa and the percent elongation, 60%. When bonded under optimal conditions, the specimens failed at the parent metal (12X18H9T steel). The specimens bonded at T = 1 273 K, p_b = 14.7 MPa and t = 10 min failed at the joint, with traces of 12X13 steel left on the surface of the 12X18H9T steel. The specimens joined at T = 1 373 K, p_b = 14.7 MPa and t = 10 min failed with a tear-out of the 12X13 steel, with an appreciable grain growth in the latter. It was found out that the 12X13 steel tended to pick up hardness. Because of this, the optimally

bonded specimens were given a heat treatment which consisted of quenching in oil from 1 323 K, tempering at 923 K, and cooling in air. This treatment had removed the inadvertently acquired hardness from the 12X13 steel.

Microstructure examination revealed no lack of bonding in the joint zone in the optimally joined specimens. The rise in temperature brought about grain growth in both the 12X18H9T and the 12X13 steel. After bonding, the 12X18H9T side of the joint showed an accumulation of precipitated chromium carbides; this was attributed to the diffusion of carbon from the 12X13 steel into the 12X18H9T steel. The heat treatment of the specimens refined their grain structure and dissolved chromium carbides, and restored the original structure of the two steels.

In the fabrication of casings (each consisting of several members), the investigators [1-4] were concerned with the effect of scale-up in passing from specimens to the actual products. It was found that the increase in size and the change in geometry affected the heating conditions, temperature distribution over the cross section, and heat withdrawal from the mating surfaces. In the subsequent re-adjustment of the welding conditions, the pressing load was brought down to 4.9 MPa so as to retain the original dimensions of the workpieces. The remaining variables were left unchanged.

Bimetallic membranes [4], which act as partitions between high- and low-pressure chambers were diffusion-bonded from 12X18H9T and 14X17H2 stainless steels. The optimal joining conditions were T = 1 373 K, p_b = 6.9-7.84 MPa, t = 10 min, and $p_{vac} = 2.6 \times 10^{-3}$-$5.2 \times 10^{-3}$ Pa. After bonding, the membranes were given low tempering at 923-943 K for 1.5 h. When fabricated under optimal conditions, the joints in the membranes matched the parent metal in strength (with the 12X18H9T steel taken as reference) (Fig. 6-7). An increase in bonding temperature and pressing load brought about appreciable plastic deformation. The bench tests of the diffusion-bonded membranes demonstrated their reliable performance.

Magnetic 20Х3МВФ(ЭИ415) steel was diffusion-bonded to nonmagnetic 0Х20Н4АГ10 (НН3) and 25Х17Н4Г15АФ2 (Н2) steels, using standard specimens 20 mm in diameter and 30 mm high. The dependence of joint strength on bonding variables was investigated in the following range: T = 1 223 K, 1 273 K, 1 323 K

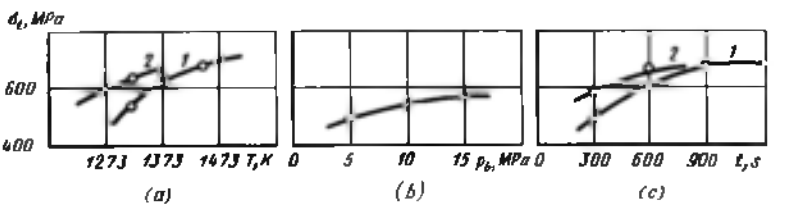


FIG. 6-7 Effect of (*a*) bonding temperature (p_b = 4.9 MPa; *1* — t = 5 min; *2* — t = 10 min), (*b*) pressing load (T = 1 373 K), and (*c*) holding time (p_b = 4.9 MPa; *1* — T = 1 273 K; *2* — T = 1 283 K) on the ultimate tensile strength, σ_t, of joints in 12X18H9T steel + + 14X17H2 steel

and 1 373 K; p_b = 4.9 MPa, 9.8 MPa and 14.7 MPa; t = 5 min, 10 min and 20 min; and p_{vac} = 0.13 Pa. Several difficulties arose because the 0Х20Н4АГ10 steel, supplied in the cold-worked condition, should retain cold working after bonding, and the recrystallization zone should be as small as practicable. On the other hand, induction heating produced structural changes on either side of the joint, with the 0Х20Н4АГ10 part recrystallizing for 10 to 15 mm from the bond zone. The hardness of this steel went down from BHN 200 to BHN 190-185. The specimens of 20Х3МВФ steel went up in hardness from BHN 190 to BHN 330-350 for a length of 16-28 mm from the bond zone. To avoid the above difficulties, it was found optimal to operate at T = 1 273 K, p_b = 14.7 MPa, t = 10 min and p_{vac} = 0.13 Pa, with the inductor shifted towards the 20Х3МВФ steel. When optimally joined, the specimens had an ultimate tensile strength of 808 MPa and a percent elongation of 40%.

For the joining of 20Х3МВФ steel to 25Х17Н4Г15АФ2 steel, the optimal conditions were found to be as follows: T = 1 373 K, p_b = 14.7 MPa, t = 10-20 min, and p_{vac} = 0.13 Pa. These bonding conditions are used in joining rotor discs for an electric motor from 20Х3МВФ and 0Х20Н4АГ10 steels, 220 mm in diameter and 28 mm thick [1, 2].

Dies used in the manufacture of fluidics components are fabricated from Р18 high-speed steel and structural steel-45. The discs are expected to retain both shape and size in service, to be strong at the joint, and to resist well up to 10 000 cycles of loading in tension and compression. Dies of intricate shape cannot be made in one piece. Fusion welding brings about prohibitive distortion. Brazing does not produce sufficient strength and does not retain the original geometry owing to the formation of fillets.

The feasibility of diffusion bonding for Р18 steel and steel-45 was investigated on specimens 20 mm in diameter and 30 mm long. The bonded joints were tested in tension, shear, and twisting. The optimal conditions were found to be as follows: T = 1 273 K, p_b = 19.6 MPa, t = 5 min, and $p_{vac} = 6.5 \times 10^{-2}$ Pa. The optimally joined specimens failed at the steel-45. The overall deformation of the specimens thus bonded was 5 to 5.5% which is unacceptable for dies and fluidics components. As a way of reducing deformation, a study was conducted into the effect of surface finish prior to bonding on the strength of bonded joints in Р18 steel and steel-45 (Fig. 6-8). When the surfaces had been machined to a roughness of not over 0.16 μm, the optimal bonding temperature and pressing load could be reduced to T = 1 173 K and p_b = 9.8 MPa. Unfortunately, even then the deformation was excessive (2.8-3.2%). In order to minimize it still more, use was made of interlayers made of lower melting point metals (such as copper and nickel). The interlayers were applied by electroplating and evaporation.

As tests in tension and shear showed, the highest mechanical strength of joints in Р18 steel and steel-45, with the temperature held at the lowest value of 1 073 K, could be obtained when the interlayer was of nickel. With a 2 μm nickel interlayer and the surface finished to a roughness of 0.36-0.04 μm, the optimal conditions of bonding Р18 steel to steel-45 were found to be as follows: T = 1 073 K, p_b = 0.98 MPa, t = 20 min, and $p_{vac} = 6.5 \times 10^{-2}$ Pa. The specimens thus joined showed a

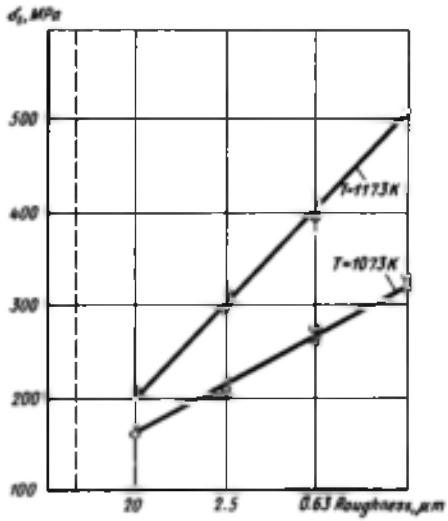


FIG. 6-8 Strength, σ_t, and percent deformation, δ%, of joints in P18 steel + steel-45 as functions of surface roughness

deformation of 0.1% and an ultimate tensile strength of 637 MPa. The shear strength was 183.3-212 MPa. Metallographic analysis of the bond zone revealed no lack of bonding. Several designs of dies have been fabricated, using the above procedure.

Diffusion bonding holds out special promise for joining 15Х18Н12С4ТЮ (ЭИ654) steel to ЭП528А alloy. One of the difficulties in this case is the increased deformability of the latter. In making diffusion bonds, special care must be taken to give an appropriate surface finish to the workpieces and to maintain the bonding temperature in proper relationship to the vacuum in the process chamber. In order to reduce the magnitude of plastic deformation in the bond zone, the bonding temperature must be kept to a minimum (not over the recrystallization point), and this can only be done by using a lower vacuum. This requirement stems from the fact that in vacuum the physico-mechanical properties of the surface layer are changed and the shear strength is reduced. As a result, the overall deformation can be brought down by a substantial amount (with a thoroughly prepared surface, the deformation will not exceed 0.6%). As the bonding temperature is reduced, surface diffusion comes to play a more important role.

For a strong and sound bond to be made between 15Х18Н12С4ТЮ (ЭИ654) steel and ЭП528А alloy, the workpieces should undergo a far smaller deformation than can be ensured on diffusion bonding machines operating at a vacuum of 0.13 Pa. Therefore, it appears attractive to carry out the bonding operation in several steps. At first, the workpieces are heated to 1373-1423 K and held at a bonding force of 49 MPa for 10-15 s. Then, while maintaining the above temperature, the bonding force is reduced to 4.9 MPa, and the workpieces are held for 15 min. During the first step, any asperities are crushed by plastic deformation at elevated temperature (while the holding time and the bonding force are minimal). The joint thus obtained

still lacks strength. Therefore, as the second step, the joint is held for another 15 min at a temperature of 973 K to produce a strong bond with minimal deformation. No noticeable structural changes can be seen in the bond zone. The bond is formed over practically all the surface. From micrographic studies, it may be concluded that, in addition to the oxide film, bond formation is handicapped by the presence of an appreciable layer of chromium carbide at the interface.

In the fabrication of automotive transmission gearboxes the need arose to make precision bonds between gear hubs and ring gears. The ring gears were made of low-carbon steel with 0.05-0.15% C, whereas the gear hubs were made of low-alloy steel with 0.16-15% C. Prior to bonding, the gear hubs had a Brinell hardness number of 140, and the ring gears, a Brinell hardness number of 100-110. In bonding, an axial pressing force was applied to the workpieces by means of a ceramic fixture. Heat was supplied by a two-turn inductor placed closer to the gear on the hub. The temperature was measured with a Chromel-Alumel thermocouple.

When gear hubs and ring gears were joined under standard conditions adopted for carbon steels, namely at $T = 1273\text{-}1373$ K, $p_b = 19.6$ MPa and $t = 5\text{-}10$ min, the deformation in the bond zone, although small, was noticeable to visual observation. To minimize the deformation, it was proposed to use two procedures each consisting of several steps.

Procedure I: The workpieces were heated to 1273-1323 K and held at a bonding force of 14.7 MPa for 5 min; then the temperature was reduced to 1 023 K, the pressing load was raised to 19.6 MPa, and the weldment was thus held for another 10 min.

Procedure II: The workpieces were heated to 1323-1373 K and held at a pressing load of 14.7 MPa for 5 s; then, with the temperature held constant, the pressing load was reduced to 8.8-9.8 MPa, and the weldment was held for another 5 min. After that, as in procedure I, the temperature was lowered to 1 023 K, the pressing load was raised to 19.6 MPa, and the parts were held for 10 min.

In either procedure, the objective of the first step was to crush asperities on the mating surfaces so as to assure a maximum area of contact. This step was instantaneous, since otherwise an appreciable deformation would have developed. The objective of the second step which was carried out at a lower temperature so as to avoid straining the weldment (at the adopted pressing load) was to promote diffusion processes. Cooling to 323-373 K in either case had as its objective to give the weldments an attractive appearance, with as few traces of oxidation as possible. After the bonding operation, the whole lot was subjected to a twist test. The hardness remained the same as it had been prior to joining. The dimensions of the parts had changed by less than 0.3 mm which was beyond visibility to an unaided eye. Macro- and microsections demonstrated that the two parts had intergrown over an annulus 0.8-1.0 mm wide.

Bibliography

1. KAZAKOV, N. F. *Diffusion Bonding in Vacuum.* Mashinostroyeniye, Moscow, 1968 (in Russian).

2. KAZAKOV, N. F. *Diffusion Bonding of Materials*. Mashinostroyeniye, Moscow, 1976 (in Russian).
3. KAZAKOV, N. F. *On Bonding in Diffusion Welding*. "Svarochnoye proizvodstvo", No. 9, 1973, pp. 48-50 (in Russian).
4. KONYUSHKOV, G. V., KOPYLOV, Yu. N., KAZAKOV, N. F. (ed.). *Diffusion Bonding in Electronics*. Energiya, Moscow, 1974 (in Russian).
5. PATON, B. Ye. *The Welding of the Future*. In: Science and Mankind. Znaniye, Moscow, 1963, pp. 504-513 (in Russian).
6. KHRENOV, K. K. *Welding, Cutting, Brazing and Soldering of Metals*. Mashinostroyeniye, Moscow, 1973 (in Russian).

7

Bonding of Cast Iron and Cast Iron to Steel

V. N. Kazakov

Cast iron owes its popularity as a material of construction to its good castability, wear resistance, good performance under alternating loads and at elevated temperature, and the low cost of cast-iron products.

Cast iron is a multicomponent iron-carbon alloy containing over 2% C, up to 5% Si and some manganese. Its sulphur and phosphorus are usually impurities. Frequently, cast iron is alloyed with chromium, nickel, molybdenum and other elements imparting it special properties.

It is customary to class cast iron into white, gray, high-strength, and malleable. In white cast iron the carbon is chemically combined with iron to form cementite which is very hard and brittle and cannot be machined by ordinary cutting tools. In gray cast iron, the carbon is usually present in the form of graphite flakes. In terms of structure, gray cast iron may be looked upon as a steel with numerous graphite inclusions. As the graphite content increases, the structure of gray cast iron changes from pearlitic, to pearlitic-ferritic, to ferritic. In high-strength cast iron, more commonly known as nodular or ductile iron, the graphite is substantially in spherical or nodular shape. Graphite nodules only slightly reduce the strength of the metal matrix. In contrast to flaked graphite, nodular graphite is not a stress concentrator. Malleable cast iron is obtained by annealing white cast iron. In it the graphite is present as flakes. The matrix is mostly ferritic, although the pearlitic form is also made.

Cast iron is poorly weldable by traditional processes because it carries a good proportion of carbon, sulphur and phosphorus. When fusion-welded, cast iron tends to crack in the near-weld zone both during and after welding. This tendency is attributed to locked stresses and the formation of chilled zones in consequence to the burn-out of silicon. Also, the weld metal is porous due to the burn-out of carbon and the presence of dissolved gases in the metal.

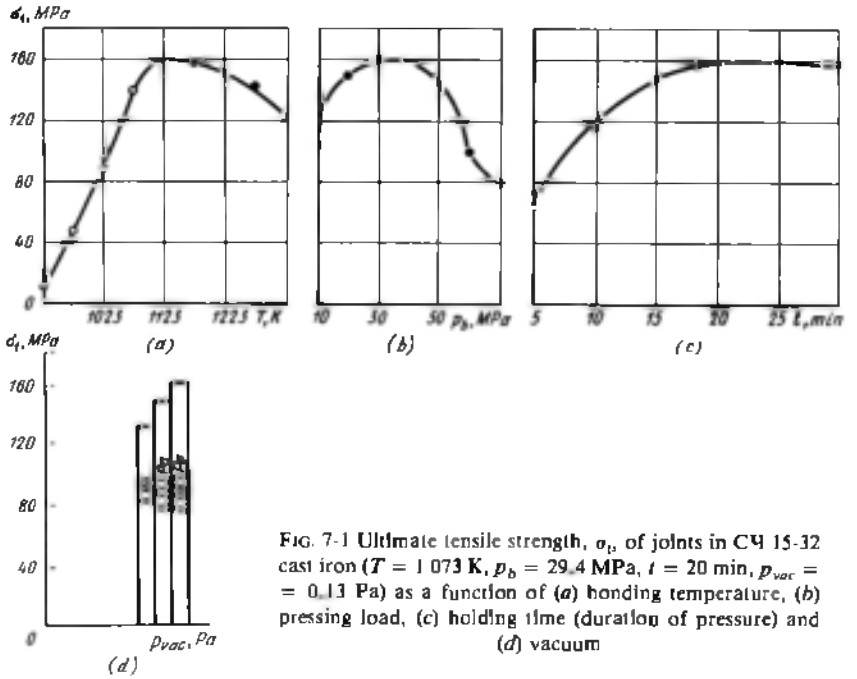


FIG. 7-1 Ultimate tensile strength, σ_t, of joints in СЧ 15-32 cast iron (T = 1 073 K, p_b = 29.4 MPa, t = 20 min, p_{vac} = = 0.13 Pa) as a function of (*a*) bonding temperature, (*b*) pressing load, (*c*) holding time (duration of pressure) and (*d*) vacuum

TABLE 7-1

Metals	Bonding variables			σ_t, MPa	a_p, MPa m
	T, K	p_b, MPa	t, min		
СЧ00 + СЧ00	1123	10	10	60	0.031
СЧ15-32 + СЧ15-32	1073	30	20	160	
СЧ15-32 + steel-45	1123	15	5	150	0.04
СЧ15-32 + steel 12Х18Н9Т	1173	15	10	165	—
СЧ15-32 + steel 14Х17Н2	1123	15	15	150	—
СЧ21-40 + СЧ21-40	1123	15	5	214	0.048
СЧ21-40 + steel-50	1173	15	5	224	0.044
КЧ30-6 + steel 12Х18Н9Т	1173	30	7	325	—
ЧНМХ[1] + steel-10	1173	20	5	—	—

[1] Composition: Ni, 0.8-1.4%; Mo, 0.2-0.5%; Cr, 0.15-0.45%.

Diffusion bonding of cast iron and of cast iron to steel has proved attractive in the fabrication of control units for hydraulic systems operating at a gauge pressure of up to 30 Pa, switches for overhead rail tracks, brake sectors, and various boiler

and pipework valves. The applicable bonding variables and the properties of the resultant bonded joints are listed in Table 7-1 and in Fig. 7-1.

Diffusion bonding is carried out at a vacuum of not over 1.33×10^{-1} Pa or in a melt of $BaCl_2$ + KCl. The roughness of the mating parts is not over 1.25-2.5 μm. Prior to fit-up, the surfaces are degreased.

In joining СЧ00 cast iron to Ст3 steel, the bond shows a diffusive carbon layer as a pearlite band 0.15-0.125 mm thick on the steel side and a decarburized layer 0.02-0.025 mm thick on the cast-iron side. No decarburized layer develops at a bonding temperature of 1 023 K.

In joining СЧ21-40 cast iron to steel-50, the interface is not visible, and the bond zone is a continuous range of solid solutions, with the carbon content increasing on its diffusion from the cast iron into the steel. The ferrite interlayers in the steel decrease in thickness on moving closer to the cast iron, and the alloy changes to a quasi-eutectoid. Then there appear graphite plates, and the structure gradually becomes that of cast iron. No chilled zone is seen.

In bonding СЧ15-32 cast iron at a temperature of over 1 123 K, the cast iron is graphitized, and an excess ferrite is formed. In the diffusion bonding of ЧНМХ cast iron under the above conditions the graphite inclusions remain unchanged.

Bibliography

1. KAZAKOV, N. F. *Diffusion Bonding in Vacuum*. Mashinostroyeniye, Moscow, 1968 (in Russian).
2. KAZAKOV, N. F. *Diffusion Bonding of Materials*. Mashinostroyeniye, Moscow, 1976 (in Russian).
3. KAZAKOV, N. F., SERGEYEV, A. V. *Diffusion Bonding of Cast Iron*. "Avtomaticheskaya svarka", 1976, No. 5, pp. 72-73 (in Russian).
4. *Welding in Mechanical Engineering. A Handbook*, Vol. 2. Mashinostroyeniye, Moscow, 1978 (in Russian).
5. STEPANOV, V. V. (ed.) *The Welder's Handbook*. 3rd edition, Mashinostroyeniye, Moscow, 1975 (in Russian).

8

Bonding of Dissimilar Metals and Alloys

V. N. Kazakov

8.1 General

Dissimilar metals and alloys are difficult to join because they differ in the physical, mechanical and chemical properties that govern the interaction between the metals and with the newly formed phases.

and pipework valves. The applicable bonding variables and the properties of the resultant bonded joints are listed in Table 7-1 and in Fig. 7-1.

Diffusion bonding is carried out at a vacuum of not over 1.33×10^{-1} Pa or in a melt of $BaCl_2$ + KCl. The roughness of the mating parts is not over 1.25-2.5 μm. Prior to fit-up, the surfaces are degreased.

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In joining СЧ21-40 cast iron to steel-50, the interface is not visible, and the bond zone is a continuous range of solid solutions, with the carbon content increasing on its diffusion from the cast iron into the steel. The ferrite interlayers in the steel decrease in thickness on moving closer to the cast iron, and the alloy changes to a quasi-eutectoid. Then there appear graphite plates, and the structure gradually becomes that of cast iron. No chilled zone is seen.

In bonding СЧ15-32 cast iron at a temperature of over 1 123 K, the cast iron is graphitized, and an excess ferrite is formed. In the diffusion bonding of ЧНМХ cast iron under the above conditions the graphite inclusions remain unchanged.

Bibliography

1. KAZAKOV, N. F. *Diffusion Bonding in Vacuum*. Mashinostroyeniye, Moscow, 1968 (in Russian).
2. KAZAKOV, N. F. *Diffusion Bonding of Materials*. Mashinostroyeniye, Moscow, 1976 (in Russian).
3. KAZAKOV, N. F., SERGEYEV, A. V. *Diffusion Bonding of Cast Iron*. "Avtomaticheskaya svarka", 1976, No. 5, pp. 72-73 (in Russian).
4. *Welding in Mechanical Engineering. A Handbook*, Vol. 2. Mashinostroyeniye, Moscow, 1978 (in Russian).
5. STEPANOV, V. V. (ed.) *The Welder's Handbook*. 3rd edition, Mashinostroyeniye, Moscow, 1975 (in Russian).

8

Bonding of Dissimilar Metals and Alloys

V. N. Kazakov

8.1 General

Dissimilar metals and alloys are difficult to join because they differ in the physical, mechanical and chemical properties that govern the interaction between the metals and with the newly formed phases.

Most pairs of metals and alloys being joined differ in melting point, density, temperature coefficient of linear expansion, lattice form, and lattice constants. The refractory and chemically active metals titanium, niobium, tantalum and molybdenum, when heated, eagerly react with nitrogen and oxygen (at a temperature of over 873 K), and this impairs their properties. These metals, their alloys and steels must be joined in a vacuum of at least 6.7×10^{-3} Pa. Copper (when free from oxygen), niobium and molybdenum must be annealed immediately before joining in hydrogen at 873 K, 1 673 K and 1 173 K for 30 min, 20 min and 10 min, respectively, whereas НП1 nickel and 29НК (Kovar) alloy must be annealed at 1 123 K and 1 073 K for 15 min and 30 min, respectively.

For weldments from metals (or alloys) sharply differing in temperature coefficient of linear expansion, the rate of cooling must be not over 15 K min^{-1}.

8.2 Bonding of Aluminium and Aluminium Alloys to Non-Ferrous Metals, Their Alloys, and Steels

Studies into the joining of aluminium to other metals have shown that the principal difficulties lie in the high chemical activity of aluminium. It reacts with other metals to form brittle solid compounds (aluminides), and with oxygen to form strong solid oxide films. The transition zone of the bond shows interlayers of aluminides and undispersed oxides which are responsible for a marked reduction in strength and impact toughness of the joints and for a large spread in their mechanical properties. It is especially important to treat aluminium and its alloys chemically prior to bonding. The surface oxide film can be removed by pickling in a solution of KOH in the case of aluminium, or in a solution of orthophosphoric acid in the case Al-Mg (АМг) and Al-Mn (АМц) alloys, followed by brightening in nitric acid. Alternatively this can be done scratch-brushing in air or in a vacuum chamber. It will be a good plan to give the surfaces thus treated a coat of acrylic resins, styrene-based varnishes and polymers, so as to prevent re-oxidation. In the course of bonding, these coatings will readily be decomposed without any residues by heating in vacuum.

Where the mating surfaces of aluminium pieces are to be electroplated with chromium, copper, silver, zinc or nickel, it will be well advised to connect the aluminium as anode in the electroplating cell, so as to dissolve its surface oxides.

BONDING OF ALUMINIUM AND ALUMINIUM ALLOYS TO COPPER. The principal difficulty when joining these metals lies in the existence of hard-to-remove oxide films on the aluminium surfaces and the formation of brittle intermetallic interlayers and oxide inclusions in the bond zone. The strength of aluminium-copper joints is decided by the transition zone which may have a varying phase composition, structure and thickness and is time- and temperature-varying.

At elevated temperatures, aluminium and copper form several brittle intermetallic compounds. At 423 K there forms Al_2Cu; at 623 K it is joined by an additional layer of Al_4Cu_9, whereas at 673 K an interlayer of AlCu is produced between the Al_2Cu and Al_4Cu_9 layers. The intervening layer has the highest hardness. When it is 10 μm thick, the strength of the bond sharply decreases. The conditions for the diffusion bonding of aluminium and of some its alloys to copper along with the properties of the resultant bond are listed in Table 8-1.

TABLE 8-1

Materials	Process variables			Bond strength, MPa
	pressing load, MPa	bonding temperature, K	holding time, min	
Copper + AMr 6	7	813	25	140
Copper + AMu	7	783	15	120
Copper + A00 aluminium	10	793	10	—
Copper + duralumin	5	873	15	—

In the diffusion bonding of aluminium to copper it is advisable to use an interlayer of zinc, silver, or nickel.

BONDING OF ALUMINIUM AND ITS ALLOYS TO STEEL. In reacting with iron, aluminium forms several intermetallic compounds, such as $FeAl_3$, Fe_2Al_2, Fe_2Al_5, $FeAl_2$, FeAl, and Fe_3Al, which cause brittleness in the joint. This is the reason why a strong joint cannot be produced by directly diffusion-bonding aluminium or its alloys to steel. The magnesium in some of the aluminium alloys likewise prevents proper bonding to steel. Magnesium is practically insoluble in iron and promotes the build-up of an interlayer of brittle intermetallic compounds because it accelerates reactive diffusion [7]. A strong joint between aluminium or its alloys and steel can only be made with some interlayer. This interlayer can be applied to the steel components by electroplating, chemically, by evaporation in vacuum, or as foil. The material for the interlayer should be chosen from an appropriate phase diagram so as to predict which phases may develop in the contact area. A very thin layer of intermetallic compounds will be formed when the interlayer is zinc, copper, silver or nickel because they prevent or slow down the formation of brittle intermetallic compounds. For example, AMu alloy and АД1 aluminium can best be joined to steel-15 with a nickel interlayer that can be deposited on the steel piece over a sublayer of copper by electroplating, or chemically, or by evaporation in vacuum. In order to obtain a joint having the same tensile strength as AMu alloy (σ_t = 135 MPa) or АД1 aluminium (σ_t = 78 MPa), the bonding variables should be as given in Table 8-2. A holding time longer than given in the table impairs the strength of the joint because the Al_3Ni_2 phase widens. The same procedure holds for joining aluminium to Ст3 steel, steel-30, Armco-iron, etc.

The quality of the diffusion bond between aluminium and iron can be improved by placing, say, copper foil between the mating surfaces. As it has a higher thermal conductivity than the metals being joined, the foil promotes the formation of a more uniform temperature field in the course of joining.

Another way of improving the quality of the joints between steel and aluminium is to give the steel, say, a copper-zinc coat (4-6 μm + 30-40 μm) or a nickel-zinc coat (5-6 μm + 30-40 μm). The layer of intermetallic compounds thus produced has then

TABLE 8-2

Materials	Bonding variables			
	temperature, K	bonding force, MPa	holding time, min	vacuum, Pa
АМц + Ni + Cu + steel-15	823	14.7	2	1.33×10^{-1}
АД1 + Ni + Cu + steel-15		12.3		

a smaller thickness and a lower hardness, whereas the tensile strength of the bond increases [6]. The same purpose can be served by silver or silver-zinc interlayers.

The diffusion bonding of X5CrNi18.9 steel to AlMgSil, AlMg3 and AlMg5 alloys with the aid of a silver interlayer may be carried out in air at a temperature of 573 K. Now the aluminium does not react with the nickel or iron, and no intermetallic compounds are formed.

When АД aluminium is bonded to 12X18H10T steel at $T = 773$ K, $p_b = 7.5$ MPa, $t = 30$ min and $p_{vac} = 1.33 \times 10^{-1}$ Pa, the joint has a maximum strength (with failure occurring in the aluminium). A transition zone is formed with a width of 4-6 μm and a microhardness of 500-1400 MPa.

The strength of aluminium-steel joints is markedly affected by the alloying elements magnesium, silicon, and copper. As already noted, magnesium has a strongly adverse effect on the weldability of aluminium. As the Mg content and the holding time are increased, the strength of the joint decreases until it is zero at a holding time of 60 min. To avoid this, AMr alloys are joined to 12X18H10T steel with an interlayer of АД1 aluminium. The alloying of aluminium with silicon has a positive effect on its weldability to 12X18H10T steel. With a silicon content 3%, the strength of the joint is raised by 20 MPa; with a silicon content of 5.6%, by 30 MPa as compared with the joint between 12X18H10T steel and АД aluminium. Any further increase in the silicon content does not practically increase the strength of the joint. The alloying of aluminium with copper is especially promising. In joining 12X18H10T steel to Al-3% Cu alloy (at $T = 798$ K), the strength of the bond increases with increasing holding time. At a holding time of 60 min, the joint is stronger than it is between the steel and АД aluminium by 50-60 MPa, but failure occurs at the bond. Longer holding times are chosen in order to retard the rate of creep for Al-Cu or Al-Si alloys.

It is possible to diffusion-bond X5CrNi18.9 steel to AlMgSiCu alloy clad with AlSi12, and the same steel, but aluminium-clad prior to joining, to Al 99.5 and AlMgSi1, AlMg3 and AlMg5 alloys in vacuum at a temperature of 773 K and with a bonding force of 9.8 MPa maintained for 20 min. The ultimate tensile and shear strengths of the joints in the above combinations of materials respectively are 22, 66, 160, 143 and 130 MPa, and 6, 5, 4.5, 6.5 and 4 MPa.

Copper-plated components from Kovar can be bonded to aluminium at $T = 793$ K, $p_b = 4.9$ MPa, and $t = 15$ min.

Tubes from X10CrNiTi 18.9 steel with an outside diameter of 25 mm, a wall thickness of 2 mm and a length of 40 mm can be diffusion-bonded to tubes from AlMg3 alloy with an outside diameter of 25 mm, a wall thickness of 2.5 mm and a length of 45 mm by heating them with two halide-quartz lamps to 803 K at the rate of 80 K min^{-1}. Prior to bonding, the tube end is tapered at an angle of 15° in a steel die. The mating end of the AlMg3 tube is flared out with a die to form an inverted cone of the same angle. Then the tube ends are inserted into each other, a bonding force of up to 175 kN is applied, and, on reaching the above temperature, the joint is held for 7 min ± 15 s. Tests for bending, tension, impact toughness, twisting and hydrostatic pressure attest to the high quality of the joint; all the specimens fail at the aluminium alloy. Prior to tensile tests, the specimens are immersed four times in liquid nitrogen and heated with hot air to 573 K.

The wedge joint between a stainless steel and AMr3 and AMr6 alloys can be made, using a pure aluminium interlayer applied to the end of the aluminium-alloy tube. The aluminium interlayer improves the thermal stability and ductility of the joint.

8.3 Bonding of Aluminium and Aluminium Alloys to Titanium, Niobium and Molybdenum

The recommended conditions for this case are listed in Table 8-3. Thus bonded, the joints are vacuum-tight.

TABLE 8-3

Nos.	Materials	Bonding conditions			Joint strength, MPa
		bonding force, MPa	bonding temperature, K	holding time, min	
1	BT1-0 + A5	12	833		188
2	BT1-0 + AMц		873		190
3	BT1-0 + A5 + AMц	14.7	893	10	195
4	BT1-0 + AMr6				125
5	BT1-0 + A5 + AMr6		863		230
6	BT1 + АД1	8	873	30	100
7	Niobium + A5				180
8	Molybdenum + A5	14.7	853	10	85
9	(Mo + Ni) + A5		833		100
10	[BT1 (BT6) + Al] + A5 + AK4	5	813	5	300
11	[BT1 (BT6) + Al] + A5 + АД26	7	773	20	190

Notes. BT1 and BT6 are titanium alloys. A5, АД1 and АД26 are aluminium alloys.

Joints between titanium or niobium and aluminium remain vacuum-tight and mechanically strong after heating at 873 K for 1 h and at 773 K for 10 h. The intermetallic compound Al_3Ti begins to grow rapidly after 2 h at a temperature of 853 K and attains a thickness of 12 μm after 4 h. The vacuum tightness and tensile strength of the specimens remain unchanged.

Joints between molybdenum and aluminium lose their vacuum tightness and mechanical strength after heating at 873 K for 30 min. When the mating surfaces are nickel-plated (6-9 μm) prior to bonding, joints between molybdenum and aluminium retain vacuum tightness after heating at 873 K during 1 h.

In combinations 1, 3, 5, and 7 (see Table 8-3), failure occurs at the aluminium. The tensile strength of the joints exceeds the tensile strength of the aluminium (50-100 MPa) because in the combinations listed it is used as a soft interlayer 0.12 mm thick, and failure in tension approaches the brittle one. In joints between titanium and AMr6 alloy, with aluminium foil as the interlayer, diffusion processes add strength to the foil. This is also true of joints between BT1 or BT6 titanium alloy and AK4 and АЛ26 aluminium alloys.

8.4 Bonding of Copper and Copper Alloys to Steel

As follows from the Cu-Fe phase diagram, the diffusion of iron into copper produces ε-solid solution of limited solubility. The solubility of iron increases monotonically from 0.3-0.35% at 973 K to 2.6-2.9% at 1 273 K. The diffusion of copper into alpha-Fe also produces a solid solution of limited solubility. In the temperature range 1023-1108 K, the solubility of copper increases to 3.5%. The supersaturation of α- and ε-solid solutions produces an α + ε eutectoid at 1 108 K. In the temperature range 1108-1183 K the solubility of copper in alpha-Fe decreases, and the copper depresses the temperature of alpha-to-gamma transition in the iron. At the above temperatures, both α-solid solution of copper in iron and γ-solid solution of copper in iron can exist; the maximum solubility of copper in gamma-iron is higher than it is in alpha-iron.

The carbon of steel impairs the mutual solubility of iron and copper, whereas manganese and silicon improve it. Manganese depresses the critical point and expands the region of γ-solid solution in which copper dissolves in a larger amount, whereas silicon hardens solid-solution grains.

In bonding M1 copper to Armco-iron (T = 1 173 K, p_b = 5 MPa, and t = 20 min), the eutectoid interlayer is 2 or 3 μm wide, and the tensile strength is 200 MPa. As the width of the eutectoid increases to 4 μm and 6 μm, the tensile strength is 125 MPa and 50 MPa, respectively. Joining at higher temperatures does not promote the formation of the eutectoid phase, because the mutual solubility of the elements increases.

The optimal conditions for joining MB, MO6 and M1 copper to Armco-iron are as follows: T = 1 273 K, p_b = 10 MPa, and t = 15 min (the ultimate tensile strength is then 220 MPa and the percent elongation, 15-17%). When БрХ0.8 chromium bronze is joined to 12Х18Н10Т steel at T = 1 173 K, p_b = 10 MPa and t = 15 min, the tensile strength is 280-350 MPa and the percent elongation is

19-24%. When the same bronze is joined to 14X17H2 steel under the same conditions the joint fails at the chromium-enriched interface layer of the bronze.

Stronger joints between copper or its alloys and iron are obtained, when a nickel interlayer is used. The nickel forms a continuous range of solid solutions with iron and copper. As follows from the Fe-Ni-Cu phase diagram, the nickel substantially increases the solubility of iron in copper and of copper in iron, with the formation of solutions of limited solubility up to a temperature of 1 183 K (in alpha-iron) and forms a range of solid solutions in gamma-iron and copper at a temperature of over 1 183 K. In the temperature range 1023-1123 K, an eutectoid interlayer is produced at the interface between the iron and nickel, consisting of a mixture of solid solutions of copper in iron and of nickel and iron in copper. At temperatures of 1 173 K and 1 223 K, a continuous range of solid solutions is formed in the transition zone.

In terms of stress-rupture strength, elongation at failure, bending angle and impact strength, the optimal conditions for joining БрХ0.8 chromium bronze and ХН67МВТЮ (ЭП202) high-temperature nickel alloy are as follows: T = 1 173 K, p_b = 1 MPa, and t = 10 min. Any further increase in holding time enriches the bronze with chromium, and this brings down the impact strength of the joint [1].

The bonding conditions used to join iron and copper cannot be used to diffusion-bond БрОСН 10-2-3 lead bronze and 40Х steel, because at a temperature of 1033-1053 K the bronze surface sweats owing to the presence of lead. The structure of the bronze consists of alpha-solid solution and inclusions of the $\alpha + Cu_{31}Sn_8$ (or $\alpha + Cu_3Sn$) eutectoid; the nickel added to the bronze increases the amount of eutectoid, but does not form any new phase, remaining in solid solution. Since lead is insoluble in copper, it is present as solitary inclusions of more or less spherical shape. The bronze has a low ductility, and its melting point is about 1 123 K. Bonding a copper interlayer 1 mm thick to 40Х steel (at 1 173 K, 10 MPa and 20 min) which is then bonded to the bronze (at 1 023 K, 5 MPa, and 10 min) produces a joint which has a higher tensile strength (229 MPa), but stands up poorly to quenching. Cracking on cooling can be avoided by applying a coat of nickel (200 μm) to a copper plate, by bonding the copper plate to the steel workpiece (at 1 223 K, 10 MPa and 20 min), and finally bonding the combination with its copper side towards the bronze piece at T = 1 023 K, p_b = 5 MPa, and t = 7 min. With failure occurring at the bronze, the tensile strength is 200-220 MPa.

The diffusion bonding of БрАЖМц 10-3-1.5 aluminium bronze to 30ХГСА steel should be carried out at T = 1 123 K, p_b = 14.7 MPa and t = 5 min with the joint subsequently quenched from 1 183 K in oil. The tensile strength of the joint is 310-320 MPa [8].

A copper-nickel alloy (such as Constantan) can be joined to 12Х18Н10Т steel at a temperature of 973 K, a bonding pressure of 29 MPa and a holding time of 10 min. When not postheat-treated, the specimens fail in tension at the bond. When the specimens are heat-treated at 973 K, they fail in the steel, and those heat-treated at 1 073 K fail in the Cu-Ni alloy.

Optimal conditions for joining copper and copper alloys to some metals and alloys are summarized in Table 8-4.

TABLE 8-4

Materials	Temperature, K	Bonding pressure, MPa	Holding time, min.
МОб copper + НП1 nickel	1173	14.7	20
МОб copper + Kovar	1223	6.8	10
МОб copper + Constantan	1223	19.6	15
Constantan + Nichrome (15% Cr-60% Ni)[1]	1173	14.7	15
Copper + БрБ2 beryllium bronze	973	4.9	20
БрХ0.8 chrome bronze + Э-steel[2]	1273	10	30
Л59 brass + steel	1123	1	20
БрОС 8-12 bronze + 12ХН3А steel	1073	20	7
БрОС 10-10 bronze + steel-10	973	5	15
БрОЦС 5-5-5 bronze + 20ХНР steel	1093	4.9	10

[1] Tensile strength, 400 MPa (in the Constantan).
[2] Tensile strength, 250-300 MPa (in the bronze).

8.5 Bonding of Copper and Copper Alloys to Refractory Metals

Copper is not easy to bond to titanium because the two metals are poorly soluble in each other and there form brittle intermetallic compounds (Ti_2Cu, TiCu, Ti_2Cu_3, $TiCu_2$ and $TiCu_3$) and a low-melting-point eutectic. When joining is done without an interlayer, the optimal conditions for МОб copper to be diffusion-bonded to ВТ1 titanium alloy are as follows: T = 973 K, p_b = 6 MPa and t = 20 min. The joint thus made is less strong than the parent metals. For the joint to match the parent metals in strength a vanadium interlayer should be used. The bonding temperature should then be 993 K.

The titanium alloys ОТ4 and ВТ14 should be diffusion-bonded to М1 copper and БрХ0.8 chrome bronze, using interlayers of molybdenum or niobium 0.1-0.2 mm thick [4]. The interlayer is evaporated onto the titanium piece and annealed in vacuum at 1 673 K for 3 h prior to bonding. The tensile strength of the resultant joint and the applicable bonding conditions are listed in Table 8-5. The higher tensile strength is related to the fact that molybdenum and niobium react with titanium to form solid solutions, whereas in contact with copper they produce no brittle phases. Niobium forms limited solutions with copper. At 1 223 K, molybdenum dissolves 2.2% (1.5 wt. %) of copper [9].

In the case of a limited plastic deformation of copper, the highest strength is achieved at T = 1 223 K, p_b = 15 MPa and t = 20 min. However, direct bonds of copper with molybdenum do not have high thermal stability, although they retain

TABLE 8.5

Interlayer material	T, K	p_b, MPa	t, min	σ_t, MPa
Molybdenum (evaporated)	1223	5	30	80-115
	1253	3.5	300	190-220
Niobium (evaporated)	1223	5	30	72-105
	1253	3.5	300	190-220
Niobium (foil, 0.1 mm)	1223	5	30	93
	1253	3.5	300	220-272
No interlayer	1073	5	30	64
	1073	3.5	300	147-160

Note. The numerator applies to induction heating, and the denominator to furnace heating.

vacuum tightness. The explanation lies in the fact that the two materials differ in the coefficient of linear expansion, and this leads to stresses in the joint when it is subjected to temperature cycling. As a way of relieving stresses and expanding the bulk interaction zone, copper and molybdenum are diffusion-bonded under the same conditions, using a nickel interlayer (5-7 μm thick) electrodeposited onto the molybdenum piece. Nickel has a high mutual solubility with the two metals being joined.

Tantalum does not form any solutions or good joints with copper. The interlayer usually employed in joining them is heat-treated БрБ2 beryllium bronze.

Direct joints between copper and tungsten are of low strength. When nickel is used as an interlayer, the optimal bonding conditions are T = 1 223 K, p_b = 16 MPa and t = 25 min. With them, the joint has a strength of 133 MPa.

The above listed bonding conditions do not produce strong copper-tungsten and copper-molybdenum weldments, they fail when subjected to not over 20 000 thermal cycles.

In order to relieve the locked stresses produced by the difference in coefficient of linear expansion and leading to cracks in tungsten and molybdenum, it is usual to make diffusion bonds at a temperature of 973 K for tungsten and a temperature of 1 073 K for molybdenum, under a pressing load of 15 MPa held for 15 min. The tungsten and molybdenum pieces are buttered with a nickel interlayer 10-15 μm thick. The shear strength of the joints thus made is 100 MPa, and they withstand 27 000 thermal cycles without failure.

In joining Л59 cast brass to zirconium, the brass piece should be treated in a solution of sulphuric acid prior to bonding so as to prevent the evaporation of zinc. The optimal conditions for joining this combination are T = 1 123 K, p_b = 5 MPa, and t = 30 min.

8.6 Bonding of Steel to Titanium

At a temperature of over 1 073 K an interlayer of intermetallic compound TiFe readily builds up at the interface between the titanium and the steel, thereby causing embrittlement of the joint. The strength of the joint is less than 50% of that of the parent metals. Titanium makes satisfactory joints with vanadium, niobium, molybdenum, zirconium and hafnium, with which it forms a continuous series of solid solutions. At a temperature of over 823 K, however, vanadium reacts with steel to form a carbide which embrittles the joint. This embrittlement can be avoided by placing a copper interlayer between the vanadium and steel pieces. Carbon does not diffuse through hard copper, and no low-melting-point eutectics are formed in the vanadium-copper system. With a small relative thickness of the copper and vanadium layers, the joint has the same strength as the stainless steel [1].

For better results, it will be a good plan to use inserts from composite V + Cu + Ni ribbon produced by rolling in vacuum to an overall reduction of 55-60% at a temperature of 1073-1173 K. The nickel layer improves ductility at the steel interface and, if kept to a small thickness, does not impair the strength of the joint. Joints in BT5-1 and AT3 alloys, bonded at $T = 1\,273$ K, $p_b = 5$ MPa and $t = 15$ min to 12X18H10T steel with a V + Cu + Ni insert have a tensile strength of 500-590 MPa.

When OT4 titanium alloy is directly bonded to 12X18H10T steel (at $T = 1\,123$ K, $p_b = 10$ MPa and $t = 13$ min) or to X22H6T steel (at $T = 1\,073$ K, $p_b = 25$ MPa and $t = 23$ min), the strength of the joint is 420 MPa and 600 MPa, respectively [1].

In joining titanium to steel, use may be made of bimetallic interlayers consisting of commercial tantalum and heat-treated БрБ2 beryllium bronze, niobium, or nonheat-treated БрБ2 bronze.

In diffusion-bonding XCr10NiTi18.9 steel to titanium, it is good practice to use interlayers as foil from vanadium and 80Cr20Ni alloy or vanadium and Ni15Cr7Fe alloy. In joining titanium to low-carbon and carbon steels, the formation of a brittle phase can be prevented by using silver interlayers.

8.7 Bonding of Nickel and Steels to Refractory Metals

The bonding conditions recommended for these combinations are listed in Table 8-6.

For a joint between ХН60В alloy and ВН2АЭ niobium alloy to match the parent metal in strength, it is advisable to use Mo-Pd and Mo-W interlayers [3].

The strength, corrosion resistance and high-temperature performance of joints between corrosion-resistant steels and refractory metals can be improved by placing a tantalum interlayer between the nickel interlayer and the parent refractory metal.

In joining molybdenum to a stainless steel, the interlayer should be made of Ta-V-Ni-Cu.

Stainless steels, such as 12X18H10T, can be joined to vanadium with an interlayer of chrome bronze, such as БрХ0.8 [3].

TABLE 8-6

Materials	Temperature, K	Bonding pressure, MPa	Holding time, min
НП1 nickel + ВТ1 titanium	973	9.8	10
НП1 nickel + МЧ molybdenum	1273	14.7	20
НП1 nickel + ВРН tungsten	1373		
НВК nickel + FeNi alloy, Kovar, Rh	1173	6.8	20
Э steel + МЧ molybdenum	1473	4.9	10
40Х steel + Nb	1273	14.7	20
12Х13 steel + Mo			
12Х18Н10Т steel + Mo	1473	9.8	15
Kovar + Mo	1373	4.9	10

8.8 Bonding of Noble Metals to Other Metals and Alloys

The optimal bonding conditions for alloys based on noble metals are listed in Table 8-7.

The alloys of noble metals listed in Table 8-7 have the following composition:

ПлИ-10: 90% Pt + 10% Ir
ПдСр-40: 60% Pd + 40% Ag
ЗлНк-5: 95% Au + 5% Ni
ПС1: powdered silver

Silver 999.9: silver of purity 999.9 parts in a thousand.

TABLE 8-7

Materials	Temperature, K	Bonding pressure, MPa	Holding time, min	Joint strength, MPa
ПлИ-10 + Ni	1173	3.5		280
ПлИ-10 + БрОФ6.5-0.15 bronze		4		170
ПдСр-40 + БрОФ6.5-0.15 bronze	1073		15	267
ЗлНк-5 + БрОФ6.5-0.15 bronze		3.2		260
Silver 999.9 + БрОФ6.5-0.15 bronze	973	3		170
Silver 999.9 + M1 copper	1023	2.5		130
Platinum + copper	1173	5		150
ПС1 + 80% Ni-20% Cu + 0.15C steel	723	10	10	263
Pd alloy + 12Х18Н10Т steel	1123	12		560
Pt + Ti	1073	6.5		
Pt (platinum/barium) + Mo	1273	13	15	
Palladium + barium + molybdenum				

8.9 Bonding of Dissimilar Metals in Porous and Nonporous Form

Porous metals are gaining ever more ground in engineering. However their use in various components inevitably poses the problem of joining both within a component and of a given component to some other structural member. Their joining by fusion welding, brazing, or cementing can only be applied on a limited scale, because the liquid phase is driven by capillary forces into the pores and changes the porosity of the blanks. Also, in fusion welding the joint undergoes an appreciable shrinkage, and this might cause cracking.

In contrast, diffusion bonding preserves the original properties of the parent metals and produces strong joints with solid (nonporous) metals.

As compared with solid metals, the joining of porous and fibrous metals necessitates the choice of bonding conditions that would take into account the tendency towards pore shrinkage. The pressing load should not exceed the yield strength of the porous metals, since otherwise it might cause changes in the shape of the component and an appreciable pore shrinkage.

The bonding conditions for porous and fibrous metals along with the tensile strength of the joints are given in Table 8-8.

TABLE 8-8

Materials	Pressing load, MPa	Temperature, K	Holding time, min	Joint strength, MPa
Porous Ni + 12X18H10T steel	3.5	1173	25	160
Porous nickel (42% porosity) + + 12X18H10T steel		1073	20	200
Porous NiCr + X21H5T steel	2.5	1223	10	160
Porous NiCr + 12X18H10T steel		1273	30	300-420
Porous Mo + БрХ0.8 chrome bronze	5.5	1173	20	125
Fibrous Cu + БрХ0.8 bronze	2.0	1123	10	74
Fibrous Cu + XH67BMIOT steel	2.5	1223	15	72
Fibrous W + БрХ0.8 bronze	5.0	1173	25	40

The composition of some of the materials is as follows:

12X18H10T steel: 0.12C-18Cr-10Ni-Ti

X21H5T steel: 21Cr-5Ni-T

БрХ0.8 bronze: 0.8% Cr

XH67BMIOT steel: Cr-67Ni-W-Mo-Al-Ti

During tensile tests, the specimens fail outside the joint area in the case of porous metals, and in the joint area, with some fibres torn out, in the case of fibrous metals. The machining of fibrous-metal specimens prior to bonding results in the separation of the fibres and impairs the quality of the joint in the case of both fibrous

copper and tungsten. Therefore, the specimens should only be degreased with petrol and dewatered with alcohol prior to joining. Diamond grinding of the specimen ends reduces the number of torn fibres as compared with machining; the quality of bonding improves.

The surface of a porous metal is far easier to prepare for bonding, because in the "as-supplied" condition it is usually impregnated with a suitable compound (porous NiCr and Ni are impregnated with ceresin, whereas molybdenum and tungsten with copper). After machining, the filler is removed by evaporation in a vacuum. In the course of diffusion bonding, it is essential to keep a close watch on all the relevant process variables. An excessive pressing load might reduce or even completely remove porosity in the contact area. The bonding temperature must be carefully controlled even at very low pressing loads, because at elevated temperatures, when the diffusion mobility of atoms and the partial pressure of metallic vapours are high, the pores might spontaneously coagulate.

8.10 Bonding of Steel to Powdered Metals

The optimal conditions for joining steel-15 to powdered nickel (ПНЭ), powdered iron (ПЖК2М2), and powdered 80% Ni-20% Cu alloy, along with the tensile strength of the joints, are given in Table 8-9.

TABLE 8-9

Materials	Temperature, K	Pressing load, MPa	Holding time, min	Joint strength, MPa
Steel-15 + ПНЭ	1233	10	10	490
Steel-15 + ПЖК2М2	1273	11	10	250
Steel-15 + 80% Ni-20% Cu alloy	1230	10	10	470

Preparatory to bonding, the steel surface should be given electric-spark treatment.

Bibliography

1. CHARUKHINA, K. Ye., GOLOVANENKO, S. A., MASTEROV, V. A., *et al. Bimetalic Joints.* Metallurgy, Moscow, 1970 (in Russian).
2. GONSEROVSKY, F. G. *Bonding of X18H9T Steel to Vanadium.* "Avtomaticheskaya svarka", 1972, No. 4, pp. 63-65 (in Russian).
3. FESENKO, A. G., AKHMETISHIN, M. A., NICHUSHKIN, V. V., *et al. Diffusion Bonding of Nickel to Niobium and of Their Alloys with Interlayers.* "Svarochnoye proizvodstvo", 1978, No. 11, pp. 16-18 (In Russian).
4. BOBROV, G. V., DOLGOV, Yu. S., PRIVEZENTSEV, V. I., *et al. Diffusion Bonding of Titanium Alloys to Copper and Copper Alloys with Molybdenum and Niobium Interlayers.* "Svarochnoye proizvodstvo", 1968, No. 11, pp. 16-17 (In Russian).

5. KAZAKOV, N. F., USHAKOVA, S. Ye., KAZAKOV, V. N. *Diffusion Bonding in Mechanical Engineering*. Kursk, 1968 (in Russian).
6. RABKIN, D. M., RYABOV, V. R. *Bonding of Aluminium and Aluminium Alloys to Steel and Copper*. Mashinostroyeniye, Moscow, 1965 (in Russian).
7. AKULOV, A. I. (ed.). *Welding in Mechanical Engineering*, vol. 2. Mashinostroyenie, Moscow, 1978 (in Russian).
8. SERGEYEV, A. V., MASHKOVA, N. A., REMIZOV, V. E. *Diffusion Bonding of* БрАЖМц 10-3-1.5 Bronze to 30ХГСА and КВК-26 Steels. "Avtomaticheskaya svarka", 1977, No. 1, pp. 72-73 (in Russian).
9. ELLIOTT, R. P. *Constitution of Binary Alloys*. McGraw Hill, New York, 1965.

9

Bonding of Refractory and Active Metals and Their Alloys

N. F. Kazakov and *V. F. Kvasnitsky*

9.1 General

Advances in technology have spurred the use of refractory metals and their alloys owing to their high-temperature behaviour, resistance to many corrosive media, and other properties.

The refractory metals used to make welded components include the metals in Groups IV, V and VI of the Periodic Table, namely niobium, tantalum, zirconium, vanadium, titanium, molybdenum, tungsten, and some others. These metals and their alloys possess a number of common physico-chemical and engineering properties, the principal ones being high melting point, chemical activity in the liquid and solid states at elevated temperatures towards the atmospheric gases, sensitivity toward thermal factors, susceptibility to embrittlement and high-rate grain growth when heated above the recrystallization temperature. The ductility of welded joints, like that of the metals themselves depends to a marked extent on the interstitial impurity content. The solubility of oxygen, nitrogen, carbon and hydrogen in refractory materials is shown in the plot of Fig. 9-1. Reacting with gases, the refractory metals form oxides, hydrides and nitrides which bring about a strongly pronounced embrittlement of the metal. Therefore, in welding active refractory metals it is important to shield the metal as best as practicable and to keep the impurity content to an attainable minimum. In this respect, special promise is held out by vacuum diffusion bonding, as it offers the best attainable shielding of the metals against exposure

5. KAZAKOV, N. F., USHAKOVA, S. Ye., KAZAKOV, V. N. *Diffusion Bonding in Mechanical Engineering*. Kursk, 1968 (in Russian).
6. RABKIN, D. M., RYABOV, V. R. *Bonding of Aluminium and Aluminium Alloys to Steel and Copper*. Mashinostroyeniye, Moscow, 1965 (in Russian).
7. AKULOV, A. I. (ed.). *Welding in Mechanical Engineering*, vol. 2. Mashinostroyenie, Moscow, 1978 (in Russian).
8. SERGEYEV, A. V., MASHKOVA, N. A., REMIZOV, V. E. *Diffusion Bonding of* БрАЖМц 10-3-1.5 Bronze to 30ХГСА and КВК-26 Steels. "Avtomaticheskaya svarka", 1977, No. 1, pp. 72-73 (in Russian).
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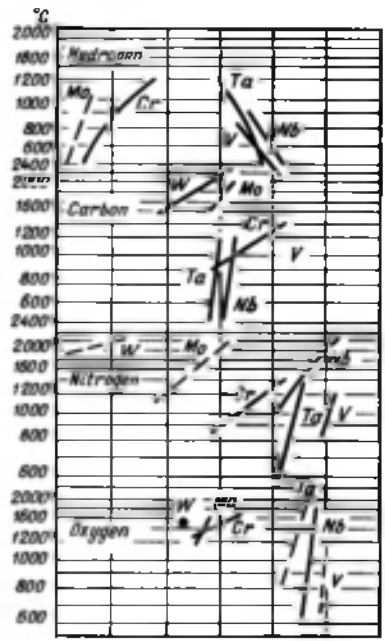

FIG. 9-1 Solubility of nitrogen, carbon, hydrogen and oxygen in refractory metals

to gases and a means for controlling the temperature and degree of deformation during the bonding cycle within the limits favourable to the metals.

9.2 Bonding of Titanium and Titanium Alloys

Titanium and its alloys offer two basic advantages over other materials. These are high specific strength (the ratio of strength to density) up to 723-773 K and good resistance to many corrosive media.

Titanium occurs in two allotropic modifications, high-temperature (beta-titanium) with a body-centred crystal lattice and low-temperature (alpha-titanium) with a hexagonal close-packed lattice. Under equilibrium conditions, the polymorphic transformation (the beta-transus) of titanium occurs at about 1 155 K. Pure titanium is used on a limited scale. According to the phase present at 293 K, titanium alloys may be classed into three groups: alpha alloys, alpha-beta alloys, and beta alloys. Those falling in the alpha group are commercially pure titanium (BT1), BT5 alloy (5% Al), BT5-1 alloy (5% Al, 2.5% Sn) and some other alloys containing alpha stabilizers. The addition of beta-stabilizers (such as molybdenum, manganese, vanadium, and chromium) to an alpha alloy turns it into an alpha-beta alloy or even a beta alloy. When the percentage of beta-stabilizers is low (not over 2%), the beta phase can only be present at elevated temperatures (this is true of martensitic titanium alloys such as OT4, OT4-1, and OT4-2). With an increase in

the percentage of beta-stabilizers, the beta phase can be present in a certain amount even at 293 K (this is true of titanium alloys such as BT6, BT6C, and BT14). Beta alloys carry so much of beta stabilizers that the only phase present even after annealing is the beta phase.

Alpha alloys have a high resistance towards embrittlement produced by the cooperative action of temperature and stress, but they have a reduced ductility. Alpha-beta and beta alloys have higher strength and respond to heat treatment, but they are less formable than alpha alloys. Also, they are susceptible to embrittlement.

Titanium and its alloys are difficult to join above all because of gas absorption, gas diffusion from the parent metal, the increased gas content in the weld zone, and structural transformations. The high activity of titanium at elevated temperatures towards oxygen, nitrogen and hydrogen reduces the ductility of the metal and causes cracking and brittle failure.

Titanium and its alloys form strong oxide films. Thermodynamic analysis and numerous experiments have shown that the most probable mechanism of removal of oxides from the surface is their dissolution in the parent metal, because titanium and its alloys, when heated, can dissolve large quantities of oxygen (up to 30% at 10^5 Pa).

Of late, there has been a growing tendency in and outside the USSR to join titanium and its alloys by vacuum diffusion bonding. For example, in the United States an assembly for a supersonic variable-geometry bomber has been diffusion-bonded from 533 titanium parts. Also in the United States, the airframe for a space vehicle capable of lifting a payload of 5.14×10^3 N has also been fabricated with diffusion bonding.

The bonding temperature is chosen anywhere between 1073-1373 K, which is within the recrystallization range and is 70-80% of the melting point. Because titanium has a low yield strength, and it is affected by temperature, the bonding pressure should be taken in the range 0.98-9.8 MPa. Depending on bonding temperature and pressure, the holding time can be anywhere from a few seconds to several tens of minutes. For example, BT5-1 single-phase titanium alloy can advantageously be bonded at a temperature of 1 273 K, a pressing load of 4.9 MPa, a vacuum of 1.33×10^{-2} Pa, and a holding time of 5 min. Bonding at temperatures not over 1 123 K, a pressing load of not over 7.8 MPa and a holding time of 1.5 min does not produce consistent results[1]. Most specimens fail at the bond, with no noticeable traces of diffusion. Bonding at that same temperature, but with the holding time extended to over 5 min produces sufficiently strong joints. The increase of bonding temperature to 1 273 K or 1 373 K seems to be sufficient, even with a holding time of 5 min, to make sound joints with a strength of 780-880 MPa, which is the same as that of the parent metal. High-strength joints have also been made with shorter holding times (1 min and 10 s), but with inconsistent results. Obviously, this value of holding time cannot be taken as optimal, at least for bonding

[1] In experiments, the vacuum was 1.33×10^{-2} Pa throughout.

temperatures in the range 1 173-1 273 K. The increase in holding time to 7-10 min has a wholesome effect on joint strength even at a bonding temperature of 1 173 K. From the results of tensile tests, it may be concluded that diffusion bonding at temperatures of 1 073 K, 1 173 K and 1 273 K for 5 min and 1 373 K for 1 min, with a pressing load of 4.9 MPa, can give ample strength and ductility matching those of the parent metal. When diffusion bonding is carried out at a temperature of 1 373 K, a pressing load of 4.9 MPa and a holding time of 5 min, there is a decrease in the mechanical properties of the joints, caused by an excessive grain growth, the alpha-to-beta-to-alpha transition on heating and cooling, and the appearance of an acicular martensitic α' structure. As tests have shown, when diffusion-bonded joints in titanium and its alloys are made under optimal conditions, they match the parent metal in impact toughness as well.

The effect of bonding temperature on the mechanical properties of joints in an alpha-beta martensitic alloy such as OT4 [3] is shown in Fig. 9-2. In the case illustrated, the bonding pressure was 0.98 MPa and the holding time was 60 min. Since, originally, the alloy had a fine-grained equiaxed structure, bonding at 1 173 K produced a bond of the same strength as the parent metal, but the specimens failed in the bond zone. As the temperature was raised to 1 198-1223 K, the tensile strength did not practically change, but failure occurred in the parent metal. The impact toughness rose sharply. At 1 223 K, it was enough to maintain the bonding pressure for 30 min. A further increase in temperature led to a bond of impaired quality: the specimens experienced brittle failure because of the coarse-grained structure and showed a low impact toughness. The effect of bonding pressure on the mechanical properties of diffusion bonds in OT4 alloy is illustrated in Fig. 9-3. As is seen, the bonding pressure is highly effective in improving the mechanical properties of the joints. The bonds made at a temperature of 1073-1123 K and a bonding pressure of 3.9-5.9 MPa match the solid metal in tensile strength, but have a low impact toughness. The increase in bonding pressure to 9.8 MPa does not raise the impact toughness of the joint to that of the parent metal. A complete analogy may be noted with the results obtained for the diffusion bonding of BT5-1 alloy. Strong joints in OT4 alloy can be made with temperatures of 1 173 K and 1 223 K and with bonding pressures of 4.9 MPa and 1.9 MPa, respectively, maintained for 30 min. The

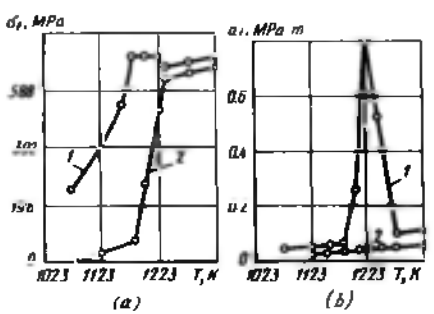

FIG. 9-2 Effect of bonding temperature on (*a*) ultimate tensile strength, σ_t, and (*b*) impact toughness, a_i, of joints in OT4 titanium alloy

1 — equiaxed fine-grained structure; *2* — lamellar coarse-grained structure

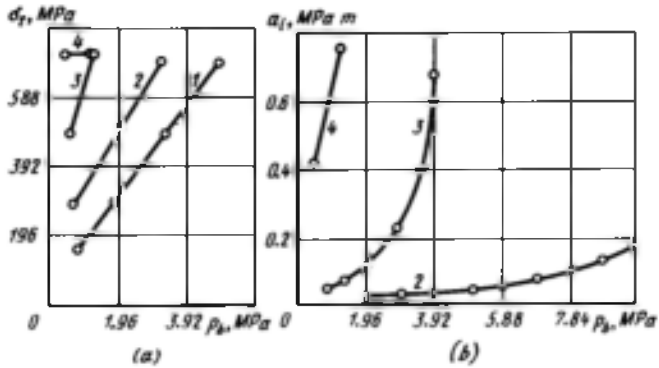

FIG. 9-3 Effect of pressing load on (*a*) ultimate tensile strength, σ_t, and (*b*) impact toughness, a_i, of joints in OT4 titanium alloy with an originally equiaxed fine-grained structure. Holding time, 30 min. Bonding temperature: *1* — 1 073 K; *2* — 1 123 K; *3* — 1 173 K; *4* — 1 223 K

specimens show a deformation of 6-8%. With the bonding pressure raised to 1.9-2.9 MPa, the bonding time is reduced to 5 min, and the deformation of the specimens is about 4%. At reduced temperatures, heavier deformation is needed in order to make strong bonds.

The distortion of weldments in titanium alloys can be minimized by using interlayers from another titanium alloy which has a higher creep rate or interlayers which will melt, be expelled or dispersed, such as those from aluminium, copper, and silver.

Special promise is held out by the diffusion bonding of titanium and titanium alloys under conditions of superplasticity. This effect substantially cuts down the bonding time, serves to make bond quality more consistent, and reduces residual deformation. In the United States diffusion bonding under superplasticity is carried out in presses capable of maintaining a deformation rate of the order of 10^{-3}-10^{-5} s^{-1} — the range where the effect of superplasticity is displayed by many materials. One US company has developed a procedure by which honeycomb members can be fabricated from titanium alloys in the following way. After fit-up and producing the desired vacuum, the members are cold-pressed under a pressure of 3.4-6.8 MPa in order to crush the asperities on the mating surfaces. Then the members are drawn apart and heated in vacuum to the desired temperature. After that, the members are again brought in contact, and the bond is made at a bonding pressure of 0.34 MPa which is sufficient and necessary for joining the skin of a honeycomb unit.

The techniques and procedures for the diffusion-bonding of titanium and titanium alloys described above are used commercially to fabricate intricate and critical structures.

9.3 Bonding of Zirconium

Zirconium occurs in two allotropic forms, the alpha form and the beta form. The alpha form exists up to a temperature of 1135-1138 K (the beta transus) and has

a hexagonal close-packed crystal structure. The beta-form is the high-temperature phase which has a body-centred cubic crystal structure. On heating, zirconium vigorously reacts with gases. It forms nitrides on reacting with nitrogen, and hydrides on reacting with hydrogen. In terms of weldability, zirconium is very close to titanium. Ordinarily, zirconium is diffusion-bonded at $T = 1\,273$ K, $p_b = 0.98$ MPa and $t = 0.5$ h, or at $T = 1023$-1223 K, $p_b = 9.8$ MPa and $t = 20$ min. Prior to bonding, the mating surfaces are machined to a roughness of 1.25 μm. When made under the first set of conditions, diffusion bonds in zirconium have an ultimate tensile strength of 528 MPa, a percent elongation of 18% and an area reduction of 12%. When made under the second set of conditions, the joints have an ultimate tensile strength of 581 MPa, a percent elongation of 20%, and an area reduction of 20%. There is a variation of the standard procedure for the diffusion bonding of zirconium. With it, the work is first heated to 1 223 K during 30 s, then held at that temperature for 30 s, then cooled below the transition point, again heated, etc. Since the two forms of zirconium differ in volume, the allotropic transformations cause strain hardening. The appearing and disappearing interfaces produce residual lattice defects which serve as sources of an excess number of vacancies. As a result, the diffusion processes proceed at a higher rate, and the diffusion coefficient increases.

9.4 Bonding of Niobium

Niobium and niobium alloys are finding an ever wider field of application owing to their high-temperature strength, corrosion resistance, ductility and formability. In most cases, niobium and niobium alloys go to make components in which permanent joints are essential.

The principal difficulties in welding niobium and niobium alloys are high activity towards gases on heating, recrystallization and embrittlement of the parent metal when heated to a high temperature. Niobium readily oxidizes, beginning at a temperature of 773 K, reacts with hydrogen at 473 K, and reacts with nitrogen to form nitrides already at 873-1073 K.

At present, a large number of niobium alloys are available. The alloying elements are molybdenum, tungsten, zirconium, vanadium, titanium, and hafnium. The addition of carbon produces niobium carbide and also the carbides of the alloying additions which add more strength and hardness to the metal. Of the niobium alloys, those most commonly used fall in a low-alloy variety, because the addition of large quantities of tungsten, molybdenum or zirconium reduces the ductility of the alloys. Examples of high-temperature niobium alloys are those made outside the Soviet Union such as B-66, D-31 and F-48, and also those of Soviet manufacture, such as BH-2 and BH-2A. A few tenths or even hundredths of one percent of interstitial elements sharply impair the ductility, deformability, corrosion resistance and weldability of the alloy.

In joining niobium and niobium alloys, the best shielding medium is vacuum. Diffusion bonds in niobium are made at $T = 1\,523$ K, $p_b = 14.7$ MPa, and $t = 5$ min. Micrographic analysis reveals no traces of interface at the bond.

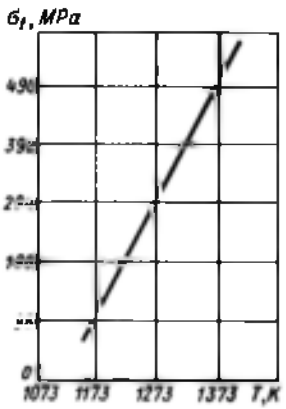

FIG. 9-4 Effect of bonding temperature on the ultimate tensile strength, σ_t, of joints in BH-3 alloy

Niobium alloys should preferably be diffusion-bonded at temperatures below the recrystallization point so as to avoid saturation of the metal with gases (O_2, H_2, and N_2) and grain growth on heating. This entails the need for a higher rate of diffusion processes through the use of interlayers applied to the mating surfaces by evaporation in vacuum. The thickness of the evaporated deposit is from several tens to several thousand angstroms. The interlayer has a very fine-grained structure. The interlayer dissolves in the parent metal, so it has no effect on the strength of the bond. In joining BH-3 niobium alloy (Mo, 4-5.2%; Zn, 0.8-2.0%; C, 0.08-0.16%; O_2, 0.03%; H_2, 0.04% max; N_2, 0.005% max; balance, Nb), the interlayer is nickel which has a low solubility in niobium and has, at a temperature of 1 373 K, a diffusion coefficient which is by three orders of magnitude lower than that of niobium in nickel. Diffusion bonding is carried out at $T = 1\ 273$ K, $p_b = 19.6$ MPa, and $t = 30$ min. Micrographic examination of the pieces with an evaporated interlayer, heated without bonding, shows that in all cases the nickel layer vaporizes from all over the surface except the zones laying at the grain boundaries. This is an indication that diffusion processes predominantly occur between the interlayer and grain boundaries on the surfaces being joined. When made with a nickel interlayer and under optimal bonding conditions ($T = 1\ 273$ K, $p_b = 19.6$ MPa and $t = 30$ min), the joint strength is 90% that of the parent metal (Fig. 9-4). No residual deformation is observed in components and specimens diffusion-bonded under optimal conditions.

9.5 Bonding of Tantalum

Tantalum eagerly absorbs nitrogen, hydrogen, oxygen, carbon monoxide and some other gases with the formation of nitrides, hydrides, oxides, carbides, and other respective compounds. Pure tantalum is highly formable and refractory. At high temperatures, however, it has a low strength. Owing to its ductility, tantalum can be alloyed with large percentages of other elements without markedly impairing the engineering properties of the alloy.

By diffusion bonding, tantalum should preferably be joined at T = 1 923 K, p_b = 11.8 MPa, and t = 20 min.

9.6 Bonding of Molybdenum

Molybdenum has a body-centred cubic lattice and undergoes no allotropic transitions up to its melting point. Molybdenum is inert towards hydrogen, resists attack by hydrochloric, sulphuric, hydrofluoric and phosphoric acids, alkali solutions and alkali-metal melts, but dissolves in nitric acid and alkali melts. It begins to react with oxygen at 673 K and to oxidize at a high rate at 873 K. Molybdenum is stable in an atmosphere of pure nitrogen from its melting point to 1 273 K. Molybdenum nitrides dissociate below 1 273 K. Commercial molybdenum alloys contain a few tenths of one percent of the alloying elements zirconium, titanium, niobium, and tantalum. When added in these amounts, they form solid solutions with molybdenum. As follows from analysis of numerous data on the diffusion bonding of molybdenum, the best results are obtained when it is carried out at T = 1 973 K, p_b = 9.8 MPa and t = 5 min. When joints are made under the above conditions, the bond zone shows no structural changes: the structure of the bond zone is similar to that of the parent metal, and there are no discontinuities in the bond. The use of fine-grained interlayers made from the parent metal has a wholesome effect on the weldability of molybdenum.

9.7 Bonding of Tungsten

Tungsten has two allotropic forms, the alpha-form and the beta-form. At a temperature below the transition temperature of 903 K, the beta-form changes to the alpha-form with a body-centred cubic crystal structure. Tungsten is stable in hydrochloric, sulphuric and other acids, and in the melts of sodium, mercury and bismuth. It does not react with nitrogen and hydrogen up to its melting point. In air it is stable up to 673 K.

Tungsten alloys are made with the addition of small amounts of niobium, zirconium, hafnium, molybdenum, tantalum, rhenium, and thoria. The primary objective of alloying is to make tungsten ductile, since at 293 K commercially pure tungsten has an elongation of close to zero. Of all the refractory metals, tungsten has the highest melting point, modulus of elasticity, and thermal conductivity, and the lowest weldability. The optimal conditions for the diffusion bonding of tungsten in vacuum are T = 2 473 K, p_b = 19.6 MPa, and t = 15 min. When so made, bonded joints closely match the parent metal in mechanical properties.

9.8 Bonding of Refractory Metals to Other Metals

Many of the design problems arising in aerospace technology, electronics, chemical engineering, shipbuilding and instrument-making can successfully be tackled by using combinations of steel and titanium or titanium alloys as structural materials. As follows from the Ti-Fe equilibrium phase diagram, the solubility of

iron in alpha-titanium is extremely low, being 0.05-0.1% at 293 K. With an iron concentration of over 0.1%, the intermetallics TiFe and $TiFe_2$ are formed in the alloy. The appearance of intermetallic compounds in a Ti-Fe alloy substantially improves strength, but sharply impairs ductility. The solubility of titanium in iron is 6.9% at a temperature of 1 573 K, but sharply goes down with decrease in temperature. At 293 K, the solubility of titanium in alpha-iron is less than 2%. The maximum solubility of iron in beta-titanium at the eutectic temperature (1 353 K) is 25%. Direct bonding of titanium to steel yields poor results. In practice, this is done with interlayers or inserts. Vanadium is the only metal that can readily be bonded to titanium and steel without the formation of intermetallics. Niobium has a lower weldability. Good results have been obtained with a combined insert made up of commercially pure tantalum (an ultimate tensile strength of 686 MPa) and heat-treated bronze. When titanium is diffusion-bonded to steels, a transition intermetallic layer is formed in the bond zone, which embrittles the joint. All in all, direct joining of titanium to steel does not assure the requisite strength and ductility in the joint.

When, in a study, BT5-1 titanium alloy was bonded to Armco iron at 973-1273K, most specimens failed at the interface in the course of machining. According to metallographic analysis of joints made at the minimum and maximum temperatures, the hardness of the metal adjacent to the interface began to increase already at $T = 970$ K, $p_b = 17.2$ MPa and $t = 10$ min. The width of the interlayer was very small (0.6-0.9 μm), so its hardness could not be measured, nor could the interlayer itself be subjected to X-ray diffractional analysis. The increase in hardness was brought about by interdiffusion of iron and titanium. When joining was done at $T = 1\ 273$ K, $p_b = 10.4$ MPa, $t = 10$ min and at the same vacuum, the intermetallic interlayer grew insignificantly, being not over 1.2 μm wide. On the iron side, however, there appeared a slightly etchable layer of increased hardness, 3 to 3.5 μm wide. This layer was responsible for the failure of joints. The ultimate tensile strength of welded specimens was 196-294 MPa. Failure occurred at the interface, without plastic deformation.

Similar results were obtained in the joining of BT5-1 alloy to X25H15 (25Cr-15Ni) steel. The use of a tantalum interlayer did not yield any positive results. Bonded joints (made at $T = 1\ 373$ K, $p_b = 11.1$ MPa and $t = 10$ min) in these two materials with a tantalum interlayer 0.08 mm thick would collapse under a slight hammer blow at the interface between the steel and the tantalum.

When tantalum is joined to steel, there appears a high-hardness interlayer presumably owing to the formation of the intermetallics Fe_2Ta and NiTa. The width of the interlayer increases with rising temperature of bonding. The Fe_2Ta interlayer is an obstacle to making a strong and ductile joint between BT5-1 titanium alloy and X25H15 (25Cr-15Ni) steel. When a nickel interlayer is, however, used, a good bond is formed between the nickel and the austenitic steel, usually accompanied by the formation of an intermetallic interlayer on the titanium alloy side.

When BT5-1 titanium alloy is joined to Armco iron with a molybdenum interlayer, the picture is not unlike that existing when the titanium alloy is joined to the steel with a tantalum interlayer. The joint fails at the interface between the iron

and molybdenum. Metallographic analysis shows the presence of a high-hardness layer between the iron and the molybdenum. On the iron side of the iron-molybdenum joint there is a slightly etchable, high-hardness band 30-50 μm wide, which results from diffusion and is a solid solution of molybdenum in iron.

In joining BT5-1 titanium alloy to Armco iron, the transition zone consists of solid solutions (on the iron and titanium sides) and the intermetallic compound TiFe. In tension, all specimens fail at the transition zone. The rupture is localized at the interface between the intermetallic compound and alpha solid solution and, in part, in the alpha phase. In the presence of a supersaturated solid solution or a wide α+ FeTi eutectoid band, failure occurs in that layer. The strength of joints is a maximum when the intermetallic layer is 3-5 μm wide.

Diffusion bonding of OT4 alloy to stainless steels can be illustrated by its joints with 12X18H10T (0.12C-18Cr-10Ni-Ti) and X22H6T (22Cr-6Ni-Ti) steels. The mechanical properties of the joints are largely the same as those of the pure metals. According to layer-by-layer spectral analysis, the interdiffusion of iron and titanium observed in the joining of OT4 alloy to a stainless steel is accompanied by the diffusion of chromium, aluminium, and nickel, with the chromium and aluminium concentrating in the transition zone. The changes in the strength and ductility of joints between titanium alloys and steels may be related to the effect of alloying, changes in the concentration of solid solutions, and the formation of intermetallics.

Joints between OT4 alloy and 12X18H10T (0.12C-18Cr-10Ni-Ti) stainless steel have an ultimate tensile strength of 412 MPa. In the case of X22H6T (22Cr-6Ni-Ti) steel the figure is 539-588 MPa.

Direct joining of BT5-1 titanium alloy to 12X18H10T steel produces bonds at most half as strong as the parent steel because an intermetallic (TiFe) layer forms.

As has been found at the Paton Electric Welding Institute in experiments with the joining of BT5-1 alloy to 12X18H10T steel, vanadium, which forms a continuous range of solid solutions with titanium and iron, is not an optimal interlayer material. This is because, in diffusion bonding, the carbon of the steel eagerly diffuses into the vanadium with the formation of vanadium carbide, VC, at the interface. The use of an additional chromium interlayer in order to suppress the diffusion of carbon owing to their interaction fails to improve the strength of joints between BT5-1 alloy and 12X18H10T steel with a vanadium interlayer. The explanation lies in the fact that the vanadium interlayer is penetrable to chromium, and this forms with titanium an intermetallic compound, $TiCr_2$. The diffusion of carbon from the steel into the vanadium can effectively be prevented with a copper interlayer. Copper does not form any intermetallic compounds with either vanadium or iron, whereas carbon does not dissolve in copper. For proper joining of BT5-1 alloy to 12X18H10T steel with vanadium or copper interlayers, the latter should be kept at some critical thickness. At critical thickness, these joints are two or three times as strong as copper and vanadium. Consistent results have been obtained with multicomponent interlayers made of V, Cu, steel and Ni and also V, Cu and Ni, by roll-bonding in vacuum at 1073-1173 K to an overall reduction of 55-60%. The nickel interlayer improves the ductility of the joint on the steel side, and the joint strength does not decrease even when the interlayer is thin.

The optimal conditions for joining BT5-1 alloy and 12X18H10T steel with a V + Cu + Ni ribbon interlayer are $T = 1\,273$ K, $p_b = 4.9$ MPa, and $t \leqslant 15$ min. For joints made with optimal interlayers the impact toughness is close to the lower limit of impact toughness for BT5-1 alloy, being 0.34 MPa. Specimens cut from the bonded bars can be bent to an angle of 50-60°. Tubular adapters bonded under optimal conditions from BT5-1 alloy and 12X18H10T steel up to 60-70 mm in diameter and 100-150 mm long show good performance in vacuum, hydrostatic, vibration and corrosion tests, sufficient for proper operation at normal and elevated temperatures.

Pieces of OT4 titanium alloy can be diffusion-bonded to 12X18H10T steel by using an interference fit as the source of bonding pressure. This technique can be used in making telescopic joints such as in the fabrication of tubular adapters. The amount of interference is chosen during fit-up so that the difference in thermal expansion and in physico-mechanical properties between the pieces being joined can produce the desired bonding pressure. The fact that the pieces are tapered in the ratio 1:100 does not markedly affect the quality of the joint because the pressing load is distributed unevenly over the mating surfaces. After fit-up, the workpieces are diffusion-bonded at $T = 1073$-1123 K, with the bonding pressure maintained for 10 min. Weldments in which the female part is made of OT4 alloy having a lower coefficient of thermal expansion than 12X18H10T steel tend to fail at the intermetallic layer due to the formation of a gap. This is an indication that tensile stresses are generated in the bond zone. In weldments with the female part made of 12X18H10T steel, the shear strength is 225-294 MPa, with the bond made under an optimal bonding pressure. Metallographic analysis of the transition zone does not reveal separation or any differences from joints made under the same bonding conditions, but with the pressing load applied externally.

The joining of titanium to copper and copper alloys by conventional methods runs into difficulties owing to a marked difference in properties, low mutual solubility, and the formation of brittle intermediate phases in the joint zone. As follows from the phase diagram, these metals produce a wide gamut of brittle intermetallic compounds, such as Ti_2Cu, $TiCu$, Ti_2Cu_3, $TiCu_2$, and $TiCu_3$, and a low-melting-point eutectic.

The best way to join titanium to copper and copper alloys is by diffusion bonding with an interlayer made from a metal which does not form brittle compounds with the parent metals. Without an interlayer, quality joints can only be made in a very narrow range of temperature and time values. In the fabrication of electron devices BT1-1 titanium alloy is joined to MB copper by diffusion bonding at $T = 1\,123$ K, $p_b = 4.9$ MPa, and $t = 10$ min. The bonding temperature is 25 degrees C below the point of formation of the liquid eutectic. The strength of bonds made without an interlayer is substantially lower than that of the parent metals, the tensile strength being 98 MPa. This procedure is used to make terminals consisting of a titanium disc and a copper washer. The components thus made fully meet service requirements.

At the PNILDSV, OT4 titanium alloy has been diffusion-bonded to БрХ0.8 chromium bronze into strong weldments. Under optimal conditions, a liquid phase

forms at the interface between the titanium and the bronze. No complete agreement with the equilibrium phase diagram for the Cu-Ti system is usually observed. In all probability, the alloying additions depress the melting point. The X-ray diffraction analysis of the transition layer reveals an intermetallic compound, Cu_3Ti, with a distorted hexagonal crystal structure. Re-heating for 5 h at 973 K sharply broadens the diffusion zone. When made under optimal conditions, bonds between OT4 alloy and BpX0.8 bronze have a tensile strength of 294 MPa. Failure ordinarily occurs at the interface between the transition layer and the OT4 alloy.

Combinations of steels and other refractory metals and of refractory metals alone are frequently utilized in engineering applications. In their properties, tantalum and niobium are very close to titanium and, when joined to it, form solid solutions without brittle compounds. Niobium can satisfactorily be joined to copper and copper alloys with which it forms limited solutions. Tantalum does not form any solutions or compounds with copper, but bronze is often used for inserts. Niobium can readily be joined to vanadium and zirconium. In joining niobium to nickel alloys, cracking is a frequent occurrence. No cracking occurs when they are joined with a palladium interlayer. The difficulties in joining refractory metals to steels and alloys are also due to the fact that the refractory metals are brittle when heated above the recrystallization point and show an increased activity when heated to over 573 K.

In joining molybdenum to 12X13 steel, metallographic analysis of the transition layer discloses zones varying in etchability. On the molybdenum side, there is a well-defined nonetchable band 0.5-3 μm wide, which has an increased hardness and brittleness. This band adjoins a wide, slightly etchable band accounting for the larger proportion of the transition zone. This zone can be etched to a varying degree which increases on moving away from the steel towards the molybdenum, and its width varies with bonding conditions. The zone is columnar in structure. The variation in etchability across the transition zone is due to the difference in the concentration of the diffusing elements and the formation of different structures. As follows from the equilibrium phase diagram for the Mo-Fe system, the two metals can form solid solutions, alpha-Fe and alpha-Mo, varying in concentration, and two intermetallic phases, epsilon-Fe_3Mo_2 and sigma-FeMo. Fe_3Mo_2 is stable at any temperature up to the melting point (1 723 K). FeMo is formed by a peritectic reaction at a temperature of 1 813 K. The sigma-phase exists in the temperature range 1453-1813 K. On cooling, this phase decomposes into the epsilon-phase and an alpha solid solution. The chromium of 12X13 steel reacts with molybdenum to form a continuous series of solid solutions. It is present in the solid solution of molybdenum and iron and is responsible for the formation of Cr-Mo ferrite. The epsilon-phase has an increased hardness and brittleness. Therefore, in making diffusion bonds between molybdenum and 12X13 steel, chromium-alloyed alpha-Mo and alpha-Fe solid solutions can be formed, and also intermetallic compounds based on Fe_3Mo_2. This is borne out by spectral and X-ray diffractional analysis. The microhardness of the molybdenum does not vary with bonding conditions, but that of the steel increases within the area of local heat build-up. This may be related to accelerated cooling upon bonding, as this brings about a martensitic transformation

in the steel. The hardness of the alpha solid solution remains the same with arying temperature and bonding pressure; that of the epsilon-phase somewhat increases with rising temperature. The joint has a maximum strength when made at T = 1 123-1 273 K, p_b = 9.8-14.7 MPa, and t = 15-20 min; its tensile strength is 382-451 MPa. This set of conditions may be taken as optimal for joining molybdenum to 12X13 steel. The reduction in joint strength with increasing bonding temperature and holding time is related to the formation of the brittle alpha-phase the hardness and width of which increase with rising temperature.

When molybdenum is joined to 12X18H9T steel, a light-coloured band is formed, which is a mixture of Mo-Fe intermetallics (such as Mo_6Fe_7) and Mo-Ni intermetallics (such as MoNi, $MoNi_3$ and $MoNi_4$). The optimal conditions for diffusion bonding are T = 1 473 K, p_b = 4.9-9.8 MPa, and t = 15 min.

The optimal conditions for bonding molybdenum to Kovar are: T = 1 373 K, p_b = 4.9 MPa, t = 10 min. Incomplete bonding is not usually observed at such conditions. The intermetallics in the joint area are distributed as finely dispersed inclusions.

Molybdenum and nickel are mutually soluble, so they can readily be diffusion-bonded. The bonding conditions are T = 1 223 K, p_b = 6.8 MPa, and t = 20 min.

Molybdenum and copper are not mutually soluble. Conflicting data have been published on joining molybdenum to copper. In some cases the joints had a sufficiently high strength of up to 157 MPa, in others their strength was unsatisfactory. A maximum strength has been achieved under conditions of limited macroplastic deformation at T = 1 223 K, p_b = 14.7-15.7 MPa, and t = 15-30 min. The joints thus made do not possess thermal endurance, but are vacuum tight. Since copper and molybdenum markedly differ in thermal expansion, stresses are generated when the workpieces are heated. The absence of a diffusion transition zone also has a negative effect, as it would otherwise promote relaxation processes in the bond. Because of this, critical Mo-Cu weldments should be diffusion-bonded with a nickel interlayer soluble in either metal. The nickel interlayer should be electrodeposited to a thickness of 7-14 μm. The best results are achieved when a single-pass nickel interlayer is deposited. A double-pass nickel interlayer or a nickel interlayer deposited over a chrome plating on the molybdenum are not satisfactory. When made under optimal conditions (T = 1223-1323 K, p_b = 14.7-15.7 MPa, and t = 10-40 min), Mo-Cu joints bonded with a nickel interlayer have a tensile strength of 148 MPa. The electric resistance of optimally bonded specimens is, on the average, $1.2 \times 10^{-6}\,\Omega$ as against $1.6 \times 10^{-6}\,\Omega$ in the case of soldered and brazed joints. The Mo-Cu joints made with nickel interlayers in components for electron devices retain a sufficient degree of strength after thermal tests (Fig. 9-5).

In an experiment, molybdenum was diffusion-bonded to ПМ30 alloy (Cu, 30%; balance Mo) in order to make a strong, permanent current-conducting joint. Specimens bonded at T = 1 223 K and t = 10 and 20 min failed during an impact test, the failure occurring at the bond. At the interface, very limited bonded areas were noticed, which indicates that diffusion had hardly had time to advance beyond the early stage. Test specimens joined at T = 1 323 K and t = 30 to 40 min fully met the requirements for mechanical strength, with failure occurring in the ПМ30

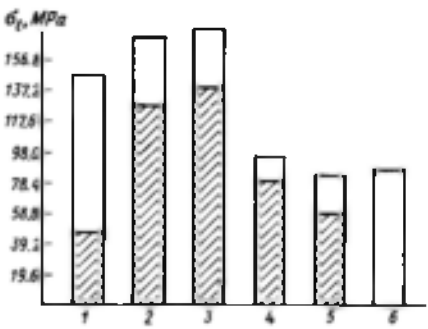

FIG. 9-5 Ultimate tensile strength, σ_t, of Mo-Cu joints in as-bonded condition (unshaded bars) and postheat-treated (shaded bars) as a function of surface preparation
1 — uncoated surfaces; *2* — single-pass nickel plating without burn-in; *3* — same with burn-in; *4* — double-pass nickel coating with burn-in; *5* — nickel plating over chromium sublayer; *6* — uncoated molybdenum; nickel-plated copper

alloy. Microscopically, the interface could be detected solely from the difference in structure between the metals being joined, which is an indication of the high quality of the joint. The optimal conditions for joining molybdenum to ПМ30 alloy have been found to be T = 1 323 K, p_b = 9.8 MPa, and t = 30-40 min.

In another experiment, tungsten was diffusion-bonded to copper directly and with an electrodeposited nickel interlayer 10-14 μm thick. The joints made with no interlayer had a low strength. Joining with a nickel interlayer under optimal conditions (T = 1 223 K, p_b = 15.7 MPa and t = 25-30 min) resulted in a strength of 130 MPa.

As part of a study, tungsten was diffusion-bonded to molybdenum at T = 1873-2173 K, p_b = 19.6-39.2 MPa, and t = 15-30 min. The specimens were tested for bending. The best results were obtained in bonding at T = 2 173 K, p_b = 19.6 MPa and t = 15 min, so this set of conditions could be taken as optimal. Yet, the specimens thus bonded had an insufficient strength. As a way of enhancing strength, tungsten was joined to molybdenum using foil interlayers from tantalum and molybdenum. In the case of tantalum foil 50 μm thick the joints were made at T = 2 173 K, p_b = 19.6 MPa and t = 20 min. Metallographic analysis clearly brought out the foil at the interface. The interface was wavy on the tungsten side, and sawtooth-shaped on the molybdenum side. Incomplete bonding had been caused by the oxides remaining on the tantalum surface. To avoid this occurrence, the vacuum in the process chamber was increased and the holding time was extended to 60 min. This did not change the microstructure at the interface — incomplete bonding persisted. The situation was different when tungsten was joined to molybdenum with molybdenum foil interlayers 50 μm thick. No interface could be discerned at places on the molybdenum side.

According to metallographic analysis, when tungsten was joined to molybdenum with a gas-flame sprayed molybdenum interlayer, no interface could be seen between the interlayer and the molybdenum; it could only be identified from the difference in grain size between the interlayer and the parent metal. When made at T = 1 973 K, p_b = 39.2 MPa and t = 15 min, the joints matched the parent metal in tensile strength. The joints made at T = 1 873 K, p_b = 49 MPa and t = 15

min matched the parent material in bending strength. With the temperature raised to 2 173 K, the bonding pressure could be brought down to 14.7 MPa.

The conditions for the diffusion bonding of tungsten-rhenium alloys to molybdenum only slightly differ from those specified for joining tungsten to molybdenum (T = 1 973 K, p_b = 34 MPa, and t = 15 min). Photomicrographs, however, distinctly show a dark band at the interface between the W-Rh alloy and the molybdenum. With all other conditions remaining unchanged, doubling the bonding pressure does not remove inclusions at the interface.

A very promising approach to joining refractory metals is the use of interlayers which promote the interdiffusion of the metals being joined. In a relevant study, tungsten was joined to molybdenum with a tantalum foil interlayer at T = 2 173 K, p_b = 19.6 MPa and t = 20 min. The joint was readily formable and could be pressed to a taper of 15° at a temperature of 1 673 K. With all other conditions held unchanged, the increase in the holding time to 60 min did not change the properties of the bond. Metallographic analysis revealed an interface between the well-defined foil and the parent metals. As an alternative, the two metals were joined at T = 2 173 K, p_b = 39.2 MPa and t = 15 min, using a molybdenum foil. Metallographic examination of the W-Mo joint disclosed a well-defined boundary between the tungsten and the foil, but no discontinuities were observed. On the tungsten side, no interface could be detected at places, and the molybdenum showed a coarse grain. Although the interface between the tungsten and the foil was clearly defined, its thickness did not differ markedly from that of the intergranular boundaries in the tungsten. This set of conditions failed to produce joints of consistent quality. When the holding time was extended to 30 min, no interface could be noticed between the tungsten and the foil; it could only be identified from the difference in grain size.

The same metals were joined with a gas-flame-sprayed molybdenum-alloy interlayer at T = 2 173 K, p_b = 39.2 MPa and t = 30 min. Metallographic analysis revealed no sprayed deposit. On the molybdenum side no interface could be discerned, whereas on the tungsten side it could only be identified from the difference in grain size between the two metals.

In joining a tungsten-rhenium alloy to molybdenum at T = 2 173 K, p_b = 19.6 MPa and t = 30 min, an electrodeposited rhenium interlayer was used. The specimen passed a formability test in a die without separation at a temperature of 1 673 K. The electrodeposited rhenium interlayer could not be detected by metallographic analysis. The interface could not be seen, and the joint showed good performance.

In the above study, the best joints between molybdenum and tungsten were made with molybdenum foil up to 50 μm thick, and those between tungsten-rhenium alloy and molybdenum with an electrodeposited rhenium interlayer.

Diffusion bonding was tried in applying niobium tips to 40X steel ultrasonic transducers operating in a molten metal. A major difficulty was the limited solubility of the two metals and a marked difference in melting point. The bonding conditions were T = 1 273 K, p_b = 14.7 MPa and t = 20 min. Metallographic analysis disclosed the presence of a thin intermetallic layer about 4 μm thick, which apparently was the epsilon-phase of $NbFe_2$.

TABLE 9-1

Material	Bonding temperature, K	Bonding pressure, MPa	Holding time, min
BT5-1 titanium alloy	1123-1273	4.9	5-10
OT4 titanium alloy	1173-1223	4.9-1.9	30
Zirconium	1023-1223 cyclically	0.9	20
Niobium	1523	14.7	5
BH-3 niobium alloy with nickel interlayer	1273	19.6	30
Tantalum	1923	11.8	20
Molybdenum	1973	39.2	15
Tungsten	2473	19.6	15
BT5-1 titanium alloy to 12X18H10T steel (with ribbon interlayer)	1273	4.9	15
Molybdenum to 12X18H9T steel	1473	4.9-9.8	15
Molybdenum to 20X13 steel	1173-1273	14.7-9.8	15-20
Molybdenum to Kovar	1373	4.9	10
Molybdenum to nickel	1223	6.8	20
Molybdenum to copper (with nickel interlayer)	1223-1323	14.7	10-40
Molybdenum to ПМ30 alloy	1323	9.8	30-40
Tungsten to copper (with nickel interlayer)	1223	15.7	30
Tungsten to molybdenum (directly or with tantalum interlayer)	1973-2173	39.2-19.6	15-60
Niobium to 40X steel	1273	14.7	20
Niobium to molybdenum	1673	9.8	5

Note. The vacuum is 1.33×10^{-2} Pa.

Specimens of niobium and molybdenum were joined at $T = 1473$-1673 K, $p_b = 9.8$ MPa and $t = 5$ min. Under metallographic analysis, the specimens bonded at 1 573 K showed a well-defined interface between the niobium and the molybdenum. In the bonds made at 1 673 K, the interface had dispersed. The optimal bonding conditions for joining niobium to molybdenum seem to be $T = 1\,673$ K, $p_b = 9.8$ MPa and $t = 5$ min.

As we have seen, the bonding conditions for joining refractory metals to one another and to other metals vary between broad limits. Suggested values of the bonding variables are given in Table 9-1.

Bibliography

1. *Records of Science and Technology. Welding,* vol. 2. VINITI, Moscow, 1979 (in Russian).

2. KAZAKOV, N. F. *Diffusion Bonding of Materials*. Mashinostroyeniye, Moscow, 1976 (in Russian).
3. KARAKOZOV, E. S., ORLOVA, M. N., PESHKOV, V. V. *Diffusion Bonding of Titanium*. Metallurgiya, Moscow, 1977 (in Russian).
4. AKULOV, A. I. (ed.). *The Welding Handbook*, vol. 4. Mashinostroyeniye, Moscow, 1971 (in Russian).
5. TUMANOV, A. T., PORTNOI, K. I. (eds.). *Refractory Materials in Mechanical Engineering. A Handbook*. Mashinostroyeniye, Moscow, 1967 (in Russian).

10

Bonding of High-Temperature Alloys, Nickel and Nickel Alloys

V. F. Kvasnitsky and N. A. Mashkova

10.1 Bonding of Nickel

Nickel, a member of Group VIIIb of the Periodic Table, is a *d*-transition metal. Engineeringly, it is a very important material. Pure nickel is very strong, tough and ductile, and it remains so at low temperatures. At 20 K the ultimate tensile strength of nickel is 774 MPa, and the percent elongation is 48%.

Nickel is only moderately reactive. In resistance to corrosion, it is superior to copper and brass, and stands up to corrosive attack by seawater, neutral and alkaline solutions of salts, sulphuric, nitric, hydrochloric and carbonic acids. It is sufficiently stable in diluted organic acids and extremely stable in alkalis at any concentration. The melting point of nickel is 1 728 K. Its density is 870-890 kg m^{-3}, and its coefficient of linear expansion is 1.33×10^{-5} K^{-1}.

Nickel is widely used in the chemical industry to make apparatus, in the electronics industry to make components for electron devices (anodes, grids, and cathode bases), and in many other industries.

When multiply alloyed, nickel makes a wide range of high-temperature alloys which are the principal materials in present-day gas turbines, jet and rocket engines (as impellers, blades, rotors, etc.), and aircraft. In the electrochemical industry, cathodes are made of Monel and Constantan, which are alloys of nickel with copper and iron.

Techniques and procedures for the diffusion bonding of nickel and nickel alloys are decided by their properties and composition, notably the thermodynamic stability of the oxide film, creep strength and deformability of the metals. On heating, pure nickel forms only one oxide, NiO, which has a relatively high vapour pressure of 1.3×10^{-5}-1.3×10^{-4} Pa at 1273-1373 K. As a *d*-transition metal, nickel reacts with oxygen to form a stable chemisorbed complex. Removal of oxygen is due

to its diffusion into the bulk metal in the course of diffusion bonding. The solubility of oxygen in nickel is 0.012% at 1 473 K, and increases with decreasing temperature. As calculations show, it can take from a few tenths of a second to several seconds for an oxide film 0.005 μm thick to dissolve in nickel at 1 173-1 473 K. Therefore, oxide films are not a major obstacle in the diffusion bonding of nickel. Electrical-engineering nickel alloys, such as Monel and Constantan, additionally form thermodynamically unstable oxides which are close to nickel in other properties, so their bonding does not differ from joining nickel.

As already noted, high-temperature nickel alloys are multiply alloyed, as they contain chromium, aluminium, titanium, molybdenum, tungsten, niobium and other elements possessing a high affinity for oxygen and imparting the alloys heat resistance and high strength at high temperatures. Precisely these properties interfere with the diffusion bonding of high-temperature alloys. The presence of a very strong and tenacious oxide film high in chromium, aluminium, and titanium, is a major obstacle to diffusion. Removal of this oxide film involves complex redox reactions.

The high tensile and creep strengths are mainly imparted to high-temperature alloys by molybdenum, tungsten and other elements suppressing the diffusive mobility of atoms. This fact is also responsible for the higher recrystallization temperature as compared with ordinary steels. Obviously, all this stands in the way of making satisfactory joints by a process which depends for its efficacy on diffusion and recrystallization. In the diffusion bonding of high-temperature alloys, one has to use higher temperatures and heavier bonding pressures than in dealing with carbon and low-alloy steels. The lower limit of temperature in diffusion bonding is taken to be the point at which recrystallization and diffusion processes just begin. For most high-temperature alloys, this point is close to 1 323-1 373 K. The upper limit is assumed to be the point at which the alloys lose their hardness and strength. The likely sharp decrease in ductility with rising temperature should also be considered. A plot of ductility-vs-temperature for two nickel alloys is shown in Fig. 10-1. As is seen, the ductility sharply decreases at 1 473 K. If, in the course of

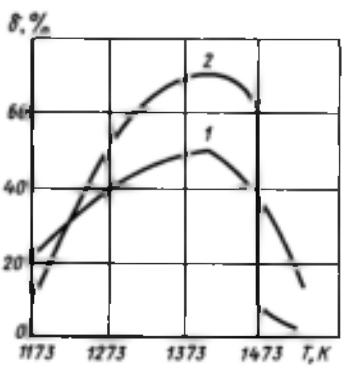

FIG. 10-1 Ductility (elongation), δ, %, of joints in (*1*) Ni-Cr-Al-Co alloy and (*2*) ЭИ 617 alloy as a function of bonding temperature

diffusion bonding, the plastic deformation is sufficient to cause the flow of the metal, cracks will develop in the bond zone. This can happen despite the low magnitude of deformation in diffusion bonding, because at elevated temperatures the plasticity is close to zero. Thus, the upper limit of bonding temperature for most high-temperature alloys does not exceed 1 473-1 523 K. The remaining process variables for the diffusion bonding of high-temperature alloys are determined as for other metals, from considerations of creep and diffusion.

Nickel to nickel and to other metals can be diffusion-bonded over a wide range of process variables. Most investigators, however, believe that this should preferably be done at a temperature of 1 273 K, a bonding pressure of 14.7 MPa, a holding time of 10 min, and a vacuum of 1.3×10^{-2} Pa. Experimental data agree well with those obtained by calculation [5] subject to the conditions required for the actual contact to be established at a steady-state creep, by the following equation

$$t = A(1/Bp_b^m)\exp(\Delta H_c/RT) \qquad (10\text{-}1)$$

where t is the time required to crush asperities, s; A is a coefficient varying with surface finish and equal to 5×10^{-3} at a surface roughness of 1.25 μm; B is a coefficient varying from 5×10^{-6} to 7×10^{-6}; p_b is the bonding pressure, MPa; m is a coefficient usually ranging between 3 and 5; ΔH_c is the activation energy for creep, kJ mol^{-1}; $R = 8.315$ kJ mol^{-1} is the universal gas constant; and T is the bonding temperature, K.

The manner in which diffusion processes occur in the bond zone in the diffusion bonding of nickel is investigated in [8]. As has been found, the diffusion flux in the bond zone undergoes variations due to changes in the quality of the mating surfaces and in the plastic deformation of the subsurface layers of the metal. In the narrow interface zone, diffusion proceeds at a very high rate (the coefficient of diffusion is 10^{-7}-10^{-8} cm^2 s^{-1}), which is an indication of a high diffusion mobility along grain boundaries and a grain misorientation favourable to diffusion. When the mating surfaces are electrolytically polished prior to bonding, the depth of the zone is 10 μm. When the mating surfaces are mechanically polished prior to bonding, the accelerated diffusion is sustained for a substantial distance from the surface. As the temperature and bonding pressure are increased, the diffusion flux becomes more homogenized over the contact area. The activation energy is about half the activation energy for the self-diffusion of nickel and is varying in the bond zone. The minimum activation energy is very close to the activation energy for grain-boundary diffusion and increases on moving away from the interfacial plane. As a number of studies have shown, the above conditions promote all the processes essential to making a quality bond with a tensile strength of up to 539 MPa. The increase in bonding temperature to 1 373 K brings about a marked grain growth owing to cumulative recrystallization. The increase in holding time leads to a loss of hardness and strength in the bond zone. It is possible to reduce the holding time somewhat, if the mating surfaces are prepared more carefully.

Nickel can be bonded not only in vacuum, but also in hydrogen. Some investigators have used hydrogen with a dew point of 233 K.

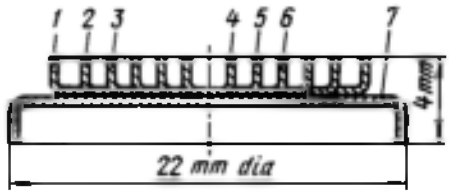

FIG. 10-2 Hydrogen-thyratron oxide cathode fabricated by diffusion bonding in vacuum

1-6 — rings; *7* — base

An example of a diffusion-bonded article from nickel is the oxide-cathode base for a thyratron, shown in Fig. 10-2.

Electrical-engineering nickel alloys, such as Monel and Constantan, are diffusion-bonded in multi-position jigs and fixtures which derive the necessary bonding pressure from the difference in thermal expansion between the work metal and the mandrel metal. Clamping bolts are usually fabricated from molybdenum. The variables for joining electrical-engineering alloys differ from those for joining nickel, as they differ in physico-mechanical properties. For example, the introduction of copper reduces the resistance of the metal to deformation, speeds up diffusion and recrystallization processes, and reduces the bonding temperature to 1 173 K.

10.2 Bonding of High-Temperature Alloys

In view of their properties and the diffusion and recrystallization processes that occur in them, the bonding temperature for high-temperature alloys is taken in the range 1 323-1 523 K. The bonding pressure and the holding time can be calculated from the desired degree of plastic deformation and the rate of creep for a given surface finish. For calculation, it is necessary to know the equation of creep for high-temperature alloys and the terms in that equation.

Depending on the bonding pressure and bonding temperature, the rate of steady-state creep for high-temperature alloys can be described by one of the following equations:

$$\dot{\varepsilon} = Bp_b^m \exp(-\Delta H_c/RT) \tag{10-2}$$

for low bonding pressures, and

$$\dot{\varepsilon} = B' \exp(\beta p_b)\exp(-\Delta H_c/RT) \tag{10-3}$$

for high bonding pressures. Here, $\dot{\varepsilon}$ is the rate of steady-state creep, s^{-1}; ΔH_c is the activation energy for creep, kJ mol^{-1}; B, B', m, and β are coefficients.

As studies have shown [11], at bonding pressures of over 49-78 MPa the creep of Nimonic alloys can best be described by Eq. (10-3). In the case of ЭИ602, ЭИ826 and ЭП99 alloys [5], the creep can best be described by Eq. (10-2), since the lower bonding pressures are more typical of diffusion bonding. In this case the constants are as follows: $B = 7.24 \times 10^{-6}$, 6.05×10^{-6}, and 5.46×10^{-6}; $m = 3.786$, 3.73, and 3.81; and the activation energy for creep $\Delta H_c = 345$, 372, and 375 kJ mol^{-1}, respectively. The constants B and m differ but little from alloy to alloy.

The activation energy for creep rises with increasing high-temperature strength of the alloys. For the precipitation-hardenable ЭП99 alloy it is higher than for the nonhardenable ЭИ602 alloy. Using the above data and Eq. (10-1), it is an easy matter to calculate the time required for the actual contact to be formed between the mating surfaces in bonding high-temperature alloys.

In joining high-temperature alloys it is extremely important to remove oxide films and to activate the mating surfaces. The dissociation, sublimation or dissolution of oxides is usually accompanied by or results from the rupture of chemical bonds, so these processes themselves could produce active centres. Unfortunately, the oxides of high-temperature alloys are thermodynamically stable and will not dissociate even in a vacuum of 1.3×10^{-2}-1.3×10^{-3} Pa. Even if they are broken down and disoriented by plastic deformation, the oxides remaining on the mating surfaces will adversely affect the performance of diffusion bonds under a sustained load at elevated temperatures. For this reason, most investigators require that the mating surfaces should be thoroughly scoured immediately prior to bonding. Some recommend that the oxides should be removed with substances which will reduce the metal from the oxide and form gaseous products of reaction. These substances include carbon, hydrocarbons, ammonium chloride and fluoride. Thermodynamic calculations and experience of vacuum metallurgy show that in vacuum the activity of carbon towards oxygen increases markedly. On reducing the metal, the carbon is oxidized to carbon monoxide which is easy to remove. It would appear that the reduction reaction could be sustained by the carbon of the alloys, but they contain a very low percentage of carbon. Therefore, carbon is applied to the surfaces in pure form or as some compounds (stearine, styrene, etc.). The amount of carbon thus applied should be carefully controlled so as to avoid any increase in the carbon concentration in the alloy being bonded. Where ammonium fluoride is used, it is decomposed by heating into nitrogen, hydrogen, and hydrogen fluoride. The oxides are removed through the formation of volatile fluorides of aluminium, silicon, titanium and other metals, and also through the reducing action of hydrogen.

As an alternative, the mating surfaces can be activated by inserting some fusible interlayers in the joint. The choice must be such that after activation the greater part of the interlayer material is expelled from the joint and the remainder dissolves in the parent metal. Some investigators have used nickel interlayers which remain solid at the bonding temperature, but improve the formability of the joint.

10.3 Bonding of High-Temperature Alloys Without Interlayers and with Solid Interlayers

In industry, diffusion bonding is widely used to join a wide range of high-temperature alloys, such as ХН75МБТЮ (ЭИ602), ХН80ТБЮА (ЭИ607А), ХН65ВМТЮ (ЭИ893), ЭП99, ЖС6К, ЖС6КП, ХН70ВМТЮ (ЭИ617), Inconel 713C, RN-41, TD-Nikel, Nimonic 90, and ХН77ТЮР (ЭИ437Б).

The optimal bonding conditions for the above cases can readily be found by calculation and through experimental design. But it is not always possible to assure

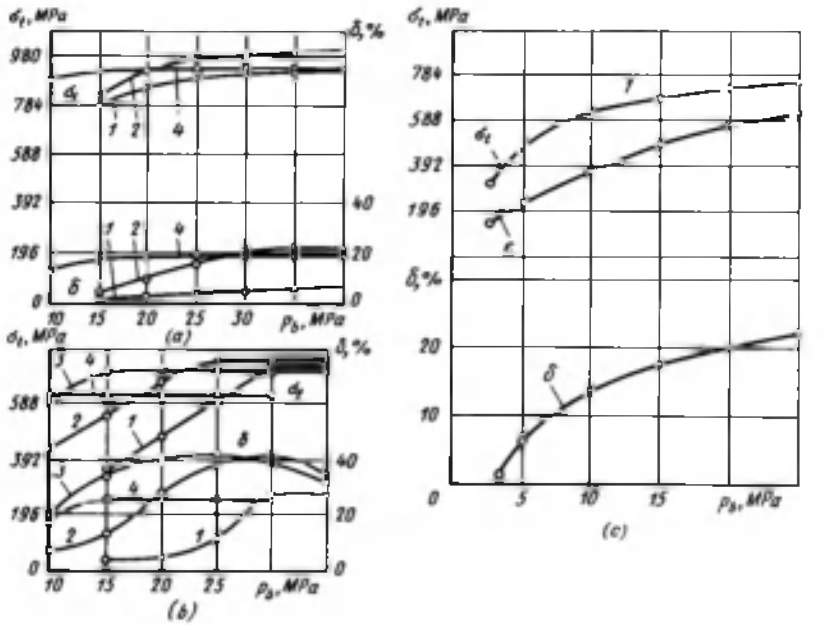

FIG. 10-3 Effect of pressing load on the properties of joints at room temperature
(*a*) ЭП 99; (*b*) ЭИ 602; (c) ВЖ 98; *1* — 1 373 K; *2* — 1 423 K; *3* — 1 448 K; *4* — 1 473 K

that the joints thus made will have properties, especially strength under dynamic loads and ductility, that match those of the parent metals. Numerous studies have shown that in the diffusion bonding of high-temperature alloys the bonding variables have the same effect on joint strength as in the case of other metals.

The effect of bonding variables on joint strength in some alloys is illustrated in Figs. 10-3 through 10-5, which also show how the deformation of specimens varies with bonding variables. In all cases, the bonding pressure was supplied by a hydraulic press. For ВЖ98 alloy the bonding pressure was maintained at constant value for the duration of the bonding operation. For ЭП99 and ЭИ602 alloys it was held constant for 4-6 s, and as it was reduced during the creep, the specimens failed to recover. This is why there was a difference in bonding pressure in joining the three alloys.

As follows from Eqs. (10-1) and (10-2), the bonding temperature, the bonding pressure, and the holding time are closely interrelated. An increase in the temperature leads to a higher rate of creep, an increased mobility of atoms, and faster diffusion and recrystallization, and this reduces the time required for the bond to form. A similar effect is produced by an increase in bonding pressure whose role can conveniently be demonstrated by reference to changes in the area of actual contact. As follows from an examination of fractographs for ЭП99 alloy, actual

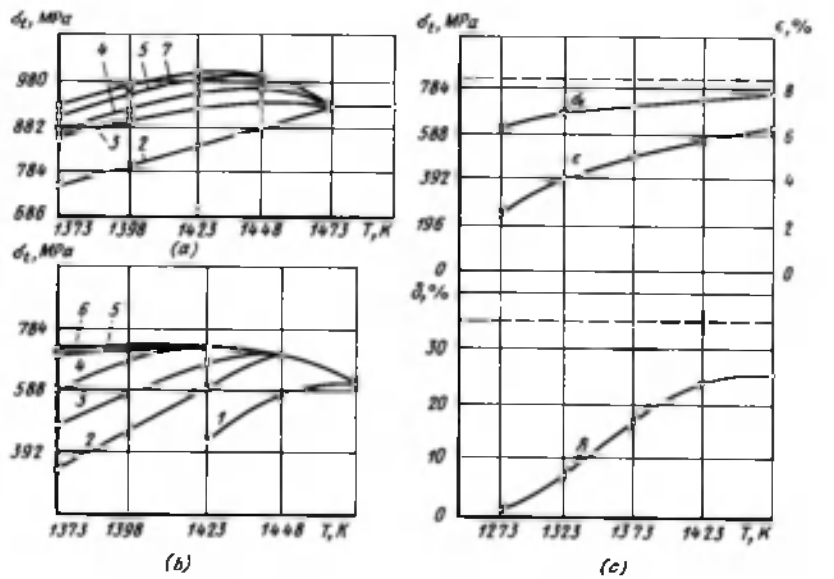

FIG. 10-4 Effect of bonding temperature on the properties of joints at room temperature (*a*) ЭП 99; (*b*) ЭИ 602; (*c*) ВЖ 98; *1* — 10 MPa; *2* — 15 MPa; *3* — 20 MPa; *4* — 25 MPa; *5* — 30 MPa; *6* — 35 MPa; *7* — 40 MPa

contact at a temperature of 1 373 K, a bonding pressure of 10 MPa and a holding time 6 min exists only over a part of the total area. The joints thus made are of low strength; there is a marked spread in ultimate tensile strength, and the percentage elongation is zero. At a bonding pressure of 15 MPa, actual contact exists over 60% of the total area, at a bonding pressure of 20 MPa, over 90% of the total area, and at 25 MPa actual contact covers the entire area. At bonding pressures in excess of 25 MPa, no microdiscontinuities are observed at the joint, but the tensile strength and, especially, ductility of the joints are low. The grain boundaries are oriented along the joint, and the specimens fail there.

Whereas joints made in ЭП99 alloy at a temperature of 1 373 K are inferior in mechanical properties to the parent metal, those in ЭИ602 alloy nearly match the parent metal in that respect. The explanation lies in the lower high-temperature strength of ЭИ602 alloy and, as a consequence, in the higher diffusive mobility of alloy atoms. In contrast to ЭП99 alloy, joints made in ЭИ602 alloy show grains which grow across the interface. The increase in bonding temperature to 1 323-1 448 K improves the strength and ductility of joints, especially those made in ЭП99 and ВЖ98 alloys. Shared grains are formed within the bond zone. The effect of bonding pressure on the properties of joints does not vary with increasing temperature, but, quite naturally, a better contact between the mating surfaces is achieved at lower

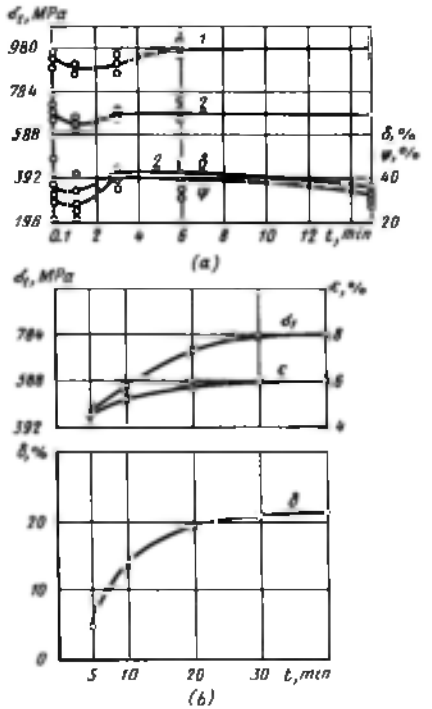

FIG. 10-5 Effect of holding time on the properties of joints at room temperature
(*a*) *1* — ЭП 99 and *2* — ЭИ 602; (*b*) ВЖ 98

bonding pressures, although the overall deformation of the specimens remains the same. For cylindrical specimens, this deformation amounts to 3%. The distribution of deformation along specimens for one of the bonding procedures is shown in Fig. 10-6. The deformation has been determined over a gauge length of 1 mm. As is seen, the deformation is a maximum at the joint and exceeds 10%. For each temperature there is a certain minimum pressure assuring proper contact between the mating surfaces within the specified time. Experimental data agree well with the

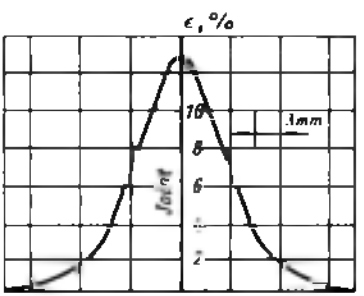

FIG. 10-6 Distribution of deformation in diffusion-bonded specimens of ЭП 99 alloy
T = 1 423 K, p_b = 40 MPa, t = 6 min

results obtained by calculation on the basis of Eq. (10-1). As the bonding temperature is raised to 1 448 K, the strength and, especially, ductility of the joints increase. Shared grains are formed across the interface within the bond zone. At a temperature of over 1 448 K, the hardening phases dissolve in most precipitation-hardenable alloys. The extent to which these phases dissolve increases with rising temperature and welding time. This entails a decrease in the strength and an increase in the ductility of the metal. The increase in bonding temperature to over 1 473 K brings about a high-rate grain growth and a reduction in mechanical properties in most alloys. At a temperature of 1 523 K, even low bonding pressures provoke cracking in many high-temperature alloys. This is why the bonding variables should be chosen with reference to the ductility diagrams.

Depending on the bonding conditions maintained, diffusion bonding in vacuum brings about a varying degree of plastic deformation in the metals being joined and entails a number of specific phenomena, such as grain twinning, subgrain formation, recrystallization, etc. Grain twinning occurs due to the cooperative action of slip along atomic planes and rotation of the deformed part of the crystal. The formation of subgrains which have a nearly perfect structure and a distinct orientation is traceable to the bonding variables. As the bonding temperature is raised, it takes a lower bonding pressure and, in consequence, a low degree of deformation to produce subgrains. As an example, in the joining of ЭП99 alloy subgrains have been observed to form at a temperature of 1 448 K, a bonding pressure of 30 MPa, and a holding time of 6 min. In ЭИ602 alloy, subgrains have been observed to form at a temperature of 1 073 K and a holding time of 8 h. In the diffusion bonding of high-temperature alloys, the grains in the bond zone are broken up into finer grains which are difficult to see at a magnification of 200 diameters, but are well observable at a magnification of 1 000 diameters. They appear along asperities on the mating surfaces, that is, within areas where the plastic deformation exceeds its average value.

The diffusion bonding of ЭП99 and ЭИ602 alloys may also be accompanied by the so-called thermomechanical treatment effect presumably responsible for the high mechanical properties of joints made with a holding time of a mere 0.1 min (Fig. 10-5). In a study concerned with the effect, the specimens were heated to a temperature of 1 323 K for 3 min, subjected to a bonding pressure of 30 MPa in the case of ЭИ602 alloy and 40 MPa in the case of ЭП99 alloy, and allowed to cool in air 6 s later. The joints thus made had relatively high mechanical properties. The increase in holding time to 1 min reduced the mechanical properties, but the further increase in holding time improved them. It is to be noted that the above bonding pressure was applied only at the start of the bonding operation, and it was not restored as it began decreasing due to plastic deformation. Because of this, holding the specimens at the resultant pressure and the subsequent recrystallization cancelled the thermomechanical treatment effect.

Thermomechanical treatment can be applied to high-temperature alloys in the following manner. To begin with, blanks are heated to 1 423-1 473 K in order to homogenize the solid solution. After they have been allowed to cool partly to 1 373 K, they are subjected to plastic deformation so as to obtain a reduction of 25-30%,

and cooled at once to prevent recrystallization. In most cases, thermomechanical treatment is followed by ageing as a way of further improving the strength and ductility of the metal.

Interestingly, this effect has been noted to persist under conditions of high-temperature plastic deformation without partial cooling and without subsequent ageing, and also at a deformation of 10% followed by quenching in water and air. Thus, it is possible to create conditions necessary for the thermomechanical effect to take place in the course of diffusion bonding, the more so that the first to deform are the asperities on the mating surfaces, and their true deformation substantially exceeds the average deformation of the specimens.

The manner in which holding time affects joint strength (Fig. 10-5*a*) can be visualized as follows. At a bonding pressure of 30 MPa for ЭИ602 alloy and 40 MPa for ЭП99 alloy, the viscous flow of the metal causes the mating surfaces to move together and interatomic bonds to form. Quenching in air at this stage may give rise to the thermomechanical treatment effect. The decisive factors are bonding pressure and temperature. The subsequent stage during which the workpieces are held at a continuously decreasing pressure results in the slow-down of metal flow, in creep at relatively low stresses, and in the onset of recrystallization. This minimizes the thermomechanical treatment effect, but leads to the disappearance of microdiscontinuities from the joint and the formation of a monolithic metal continues. With a holding time of 1 min, the impairment in the mechanical properties can be explained by the cancellation of the thermomechanical treatment effect and inadequate diffusion processes. In many experiments, the strength and ductility of joints have been improved by restoring the bonding pressure after a holding time of 1 min, followed by quenching.

What has been said about the effect of holding time on the properties of bonded joints is valid only at relatively high bonding pressures sufficient for plastic deformation to produce contact between the mating surfaces in a matter of a few seconds. The formation of such a contact is confirmed by the fact that holding the workpieces for 5 min at the bonding temperature, with bonding pressure removed within 10 s after application, results in joints which match the parent metal in strength. When the bonding pressure is 20 MPa, it has to be maintained constant for several minutes in order that actual contact between the mating surfaces can be formed due to the creep of the metal at constant stress. Similar results have been obtained for ВЖ98 alloy (Fig. 10-5*b*). The common yardstick with which to measure the effect of bonding pressure at different levels is the degree of plastic deformation of the metal. In most cases, joints will match the parent metal in strength if the deformation of the metal at the joint is 5-8%.

Proper bond formation is to a great extent dependent on the quality of the mating surfaces prior to welding. It has been found that the density and distribution of lattice defects in the subsurface layers greatly varies with the manner in which the mating surfaces are prepared (by turning, mechanical grinding or electrolytical polishing). However, these defects are not stable and can be re-distributed upon heating without stress due to their emergence on the surface and annihilation. At relatively small bonding pressures producing a plastic deformation of several tenths

of one percent, the area of actual contact depends on the number and character of asperities on the surfaces. Polishing has been found to yield the best results. When the bonding pressure is increased to values which produce a marked plastic flow of the subsurface layers (a deformation of about 10%), the number and character of asperities on the surfaces have no marked effect on the area of actual contact. In [13] it is proposed to give the mating surfaces a pattern of regularly alternating grooves and tongues as in a threaded joint. Good results have been obtained when the grooves make an included angle of 60° and are spaced 1 mm apart.

Bonding variables can be selected from thermokinetic recrystallization diagrams, such as plotted for ХН77ТЮР (ЭИ437Б) high-temperature alloy. Entering the diagram with the magnitude of preliminary plastic deformation, we can determine the bonding temperature and holding time required to complete the bond. For specimens without preliminary deformation, the performance of joints improves with rising temperature and increasing time by analogy with ЭП99 and ВЖ98 alloys.

In striving to obtain common (shared) grains in the bond zone, measures must be taken to avoid any difference in grain size between the parent metals, as this impairs both the ductility and high-temperature strength of the joint. This can be achieved by consulting the applicable recrystallization diagram so as to prevent rapid grain growth. As is seen from the recrystallization diagram for ЭП99 alloy in Fig. 10-7, the grain size is a maximum at a bonding pressure corresponding to critical deformation, past which the grain size decreases. The bonding pressure producing critical deformation decreases with rising temperature.

As follows from microanalysis of joints in ЭП99 and ЭИ602 alloys, when the bonding temperature is 1 423 K and the bonding pressure is over 30 MPa, a few seconds is enough for grains common to both pieces to be formed. Such grains take up fairly extended areas. This is an indication that common grains are formed from grains differing in orientation over a short span of time, rather than because the grains in the mating pieces coincide in crystallographic orientation by chance. The

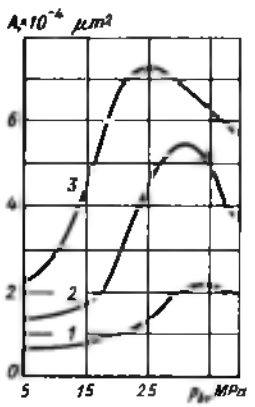

FIG. 10-7 Recrystallization diagram for ЭП 99 alloy
1 — 1 373 K; *2* — 1 448 K; *3* — 1 473 K

formation of such grains is promoted by the diffusion mechanism of plastic deformation with which the bonds lose their directivity and the grains break down. In contrast, it is impeded by the microdefects and contaminants present at grain boundaries within the bond zone. Within areas where the mating surfaces are in actual contact and the grain boundaries retain their orientation in the joint plane for a few seconds after joining, common grains can be produced by increasing the holding time without any bonding pressure applied. This is an indication that bonding pressure is most essential for the metal to be deformed and for an intimate contact to be established between the mating surfaces. If the bonding pressure is not sufficient for proper contact to be formed at the outset of bond formation (and, as a consequence, the rate of creep is low), the duration of bonding pressure becomes the decisive factor in bond formation. This underscores the need for considering the degree of deformation in diffusion bonding.

In the diffusion bonding of high-temperature alloys which produce thermodynamically strong oxide films, the degree of deformation plays an important role also in the formation of metallic bond between the mating surfaces. With properly chosen values of bonding pressure and holding time, a plastic deformation of up to 10 % can be achieved in the joint zone. As an example, for high-temperature alloys such as ХН80ТБЮА and ХН65ВМТЮ when they are joined at a bonding pressure of 20 MPa and a vacuum of 1.3×10^{-2} Pa, the recommended temperature is, respectively, 1 473 K and 1 448 K and the holding time, 6 min and 20 min. In the case of ХН80ТБЮ alloy, microanalysis of the joint reveals both grains common to the two pieces and traces of the interface. These traces in the form of microdiscontinuities and oxides reduce the ductility of the joint. In the case of ХН65ВМТЮ alloy, microanalysis reveals practically no traces of the interface. At 293 K and 1 023 K, the joints in this alloy have the same strength as the parent metal. At 293 K, the specimens fail viscously in the bond zone, whereas at 1 023 K they do so in the parent metal. The long-time (stress-rupture) strength of the joints, as measured at 1 023 K over 10^5 hours, is 144 MPa. Failure occurs viscously and takes place in the parent metal.

Joints in ХН80ТБЮ alloy, made as advised above and heat-treated at 1 273 K for 2 h followed by ageing at 1 023 K for 20 h, have a long-time strength of 127 MPa as measured at 973 K over 10^5 hours. This is 80% of the long-time strength of the parent metal.

As follows from analysis of joints made in ВЖ98, ЭП99 and ЭИ602 high-temperature alloys (see Figs. 10-3 through 10-5), they match the parent metal in strength, but they are inferior in terms of ductility. When joints made in ВЖ98 alloy at 1 273-1 473 K are additionally held at 1 473 K (Fig. 10-8), their strength changes but little, but their ductility is sharply increased, especially when the bonding temperature is below 1 373 K. When tested at temperatures in the range 973-1 173 K, the joints have a strength very close to that of the parent metal, but a lower ductility.

During high-temperature tests, butt joints made in ЭП99 and ЭИ602 alloys under optimal conditions match the parent metal in strength. The ultimate tensile strength of joints in ЭП99 alloy at 1 173 K is about 510 MPa, and that of joints in

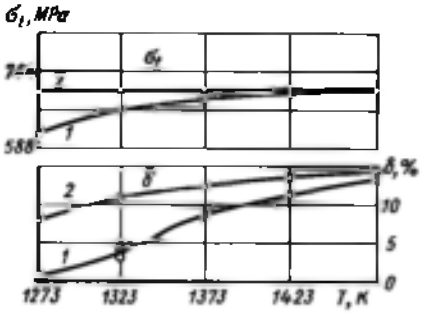

FIG. 10-8 Effect of extended diffusion treatment on the mechanical properties of joints *1* — as-bonded; *2* — after extended diffusion treatment for 20 min at 1 473 K

ЭИ602 alloy at 1 073 K is about 350 MPa. The percent elongation is, on the average, 4.8% in the former case, and 12% in the latter, which is smaller than for the parent metal.

The bondability of ЖС6КП alloy has been investigated by varying the bonding temperature in the range 1 073 -1 273 K, the bonding pressure in the range 10-30 MPa, and the holding time in the range 2-15 min. The optimal bonding conditions have been found to be as follows: temperature, 1 273 K; bonding pressure, 20 MPa; holding time, 10 min; vacuum, 1.3×10^{-2} Pa. The increase in bonding temperature to 1 373 K assures high strength, but this improvement is accompanied by a high-rate grain growth. Bonding pressure in excess of 20 MPa results in excessive deformation and cracking in the specimens.

From a study in the bondability of ЖС6 cast nickel alloy it has been learned that it calls for thermomechanical treatment. In ЖС series of alloys, full-strength joints can only be made if the permanent set is about 8%. In joining them to alloys having a lower high-temperature performance it will suffice to have a deformation of about 5%.

Where precision weldments are to be fabricated from high-temperature alloys, it is a good plan to use a nickel interlayer because it facilitates the formation of actual contact between the mating surfaces. A number of investigators both in and outside the Soviet Union [16, 17] have obtained good results with other interlayers as well. In the case of Udimet 700 alloy it was learned that several factors stood in the way of proper bonding, the most crucial one being the formation of a continuous series of intermetallic compounds Ti (C, N) and $NiTiO_3$ in the joint. These compounds tended to retain and weaken the grain boundaries in the joint zone. A thin nickel interlayer electrodeposited on the mating surfaces was sufficient to prevent the formation of the above intermetallics, but it gave rise to residual chemical and microstructural discontinuities in the metal. The microstructural and chemical homogeneity of the joint was markedly improved through the use of an electrodeposited Ni-Co interlayer containing 25-35% Co. This could be due to several factors, including the likely increase in the diffusion of the alloying elements and the reduced risk of embrittlement in the γ'-layer which formed in joints with a nickel interlayer owing to the increased diffusion of titanium and aluminium into the nickel. Specimens of Udimet 700 were diffusion-bonded at $T = 1\,443$ K, $p_b = 6.9$ MPa and $t = 4$ h, and

also at T = 1 363 K, p_b = 20 MPa and t = 2 h. The Ni-Co interlayer prevented the formation of the γ'-phase between the mating surfaces and provided for a high structural homogeneity in wrought Udimet 700 alloy upon ageing which followed bonding. In the case of cast Udimet 700 alloy the grain boundaries ran along the joint, but the joint nearly matched the parent metal in short-term strength. Since tensile tests usually fail to give an accurate idea about the performance of high-temperature alloys, specimens of Udimet 700 were creep-tested at 1 033 K and 1 253 K, and the joints showed poor performance. To improve it, it was chosen to bond cast Udimet 700 alloy at T = 1 463 K, p_b = 8.5 MPa and t = 4 h; in the specimens so bonded it was difficult to discern the interface. When creep-tested, the specimens showed performance close to that of the parent metal, whereas the service life of some specimens was even longer than that of the parent metal. When Udimet 700 was bonded without an interlayer, no satisfactory joints were made.

TD-Nikel was bonded at T = 1 366 K, p_b = 135 MPa and t = 2 h with interlayers made from various materials. The best results were obtained with a cobalt alloy interlayer 0.1 mm thick. The material was also bonded without an interlayer, and the lap joints matched the parent metal in strength. TD-Nikel is a purely nickel matrix strengthened with a finely dispersed thoria ThO_2 (around 2 vol.% thorium). It has a good ductility and structural stability up to 1 580 K. In Britain, use is also made of TD-NiCr alloy which goes to make heat shields for spacecraft. A heat shield of TD-NiCr alloy is diffusion-bonded to the main plating at a vacuum of 2.66×10^{-3} Pa.

To speed up the formation of a bond in the case of Feduska and Xorigan alloys, the interlayer included beryllium which has a small atomic radius and a high diffusive mobility. As an optimum combination, it was proposed that the interlayer should be made of 3% Be-Ni, Ni-Cr-Be and Ni-Cr-Fe-Be alloys. The diffusion bonds made at 1 333-1 423 K with the bonding pressure maintained for 0.5-5 min showed high mechanical properties very close to those of the parent metal. When bonded with a nickel interlayer, no satisfactory joints were made in the two alloys, although some investigators [15] do recommend such interlayers. They bonded Nimonic 90 Ni-Cr-Co alloy with a nickel interlayer 0.024 mm thick at a pressing temperature of 1 373 K, a pressing load of 32 MPa and a holding time of 15 min, followed by heat treatment for 20 h at 1 373 K and for 16 h at 973 K. The joints thus made had an ultimate tensile strength of 1 039-1 068 MPa at room temperature, which was 95-97% of that of the parent metal. Owing to the diffusion of the alloying elements from the parent metal, the composition of the interlayer changed across the entire thickness, but the high-temperature properties of the joints were as low as 23-51% of their values at room temperature. With a foil two times as thick, the efficiency of the joint at 293 K was one-fourth of its original value.

In the diffusion bonding of ЭИ602 alloy, a nickel foil 0.1 mm thick was used. The joints were made at T = 1 423 K, p_b = 15 MPa, and t = 6 min. Some of the specimens were tested immediately after bonding, and the remaining ones after holding them for 8 h at a temperature of 1 073 K which was expected to occur in actual service. In all cases the strength of the joints at 293 K was at least 80% of the strength of the parent metal, whereas at 1 073 K it was equal to the short-term

strength of the alloy. In either case, the strength of the joints exceeded that of nickel. The long-term strength of the joints with an interlayer proved very low. Holding for 8 h at 1 073 K had no effect on the strength of the joints.

As follows from an analysis of joints made in high-temperature alloys with interlayers, the effect of the latter should be assessed in terms of the performance of joints made with soft interlayers. In detail, this matter is examined in [1]. Taking round specimens under a static tensile load as an example, the ultimate tensile strength of the joint with allowance for the contact hardening of the interlayer material due to the containment of plastic deformation in the interlayer by the stronger parent metal, and neglecting strain hardening, is given by

$$\sigma_t = \sigma_y^i(1 + 1/3\kappa\sqrt{3}) \tag{10-4}$$

where σ_t is the ultimate tensile strength, MPa; σ_y^i is the yield strength of the soft interlayer in tension, MPa; and $\kappa = h/d$ is the ratio of the interlayer thickness to the specimen diameter (or to thickness for flat specimens), or the relative thickness of the soft interlayer.

For flat specimens

$$\sigma_t = (2\sigma_y^i/\sqrt{3})(1 + 1/4\kappa) \tag{10-5}$$

It follows from Eqs. (10-4) and (10-5) that the ultimate tensile strength of a joint is decided not only by the properties of the metal, but also by the relative thickness of the interlayer. As the relative thickness κ is decreased, the ultimate tensile strength increases. Therefore, for mechanically inhomogeneous joints to match the parent metal in strength, progressively thinner interlayers should be used.

For high-temperature alloys, the more important indicator is the performance of joints with a soft interlayer under conditions of creep. Since creep is constrained, a volumetric state of stress arises in the soft interlayer. The maximum normal stresses are directed along the axis and for plane deformation are given by

$$\sigma^i = \left(1 + \frac{1}{1+\mu}\right)p_b \tag{10-6}$$

and in the case of axisymmetric deformation they are given by

$$\sigma^i = \left(1 + \frac{2}{1+\mu}\right)p_b \tag{10-7}$$

where p_b is the bonding pressure averaged over the cross section, MPa; $\mu = 1/m$ where m is the exponent in Eq. (10-2) when creep obeys the power law. For actual values of m in the region of brittle failures, the long-term strength of the soft interlayer may be by a factor of 1.8-2.6 lower than when the deformation of the interlayer material is not constrained.

In the case of viscous failure, the long-term strength of a joint with a soft interlayer is given by

$$(\sigma_t^i)_\kappa = \sigma_t^i K_\kappa \tag{10-8}$$

where σ_l' is the long-term tensile strength of the soft interlayer under conditions of unconstrained deformation, MPa; and K_a is the coefficient of contact hardening of the soft interlayer. In the case of plane deformation it is defined as

$$K_a = (2/\sqrt{3})(1 + 1/4\kappa) \tag{10-9}$$

and in the case of axisymmetric deformation it is

$$K_a = (1 + 1/3\kappa\sqrt{3}) \tag{10-10}$$

As follows from analysis of the above equations, the joint strength will sharply decrease if the interlayer is embrittled in the course of creep. The less the interlayer material tends to grow brittle, the longer the time interval during which the interlayer will be contact-hardened.

The performance of joints with a soft interlayer can also be assessed in terms of the mechanical properties of the interlayer as compared with those of the parent metal. In considering the role of interlayers in bond formation, some investigators limit themselves to the improvement in the conditions under which bonding occurs due to the presence of a softer metal, the higher rate of diffusion processes, and the protection of the mating surfaces when the interlayer is electrodeposited. In our opinion, the shear stresses that develop at the interface between the parent metal and the interlayer are no less important. Shear stresses drive dislocations to the surface, disperse the oxide films, and activate the bonding process.

Alternatively, oxide films can be removed by exposure to ammonium fluoride, carbon, or carbon-bearing compounds. This makes the joining process less sensitive to variations in the bonding variables and improves the mechanical properties of the joint. Most conveniently, a carbon deposit can be applied by vacuum evaporation. For ЭИ437А alloy it has been found that optimally the carbon deposit should be 0.03-0.04 μm thick. Electron-diffractometric examination has shown that such a carbon deposit can effectively remove oxide films and enable the joints thus made to match the parent metal in strength. Specimens bonded without a carbon deposit or with a deposit 0.08-0.1μm thick fail through the joint. Owing to the reduction of the oxides by carbon, the bonding temperature can be brought down to 1 323-1 373 K at a pressing load of 30 MPa.

10.4 Bonding of High-Temperature Alloys with Fusible Interlayers

Frequently, diffusion bonding in vacuum is used to join high-temperature alloy components with extended plane or odd-shaped surfaces. In such cases, it is extremely difficult to assure an intimate actual contact over the entire area. In fact, extreme care in surface preparation, high pressing loads and expensive equipment may all prove wasted because the bonding pressure cannot be distributed uniformly over the contact area.

In the diffusion bonding of thin-sheet assemblies, further difficulties arise, because the pressing fixture has to be heated to the same temperature as the pieces being joined. When a jig or fixture is exposed to the bonding pressure for a long

time, its material begins to creep, and its service life is drastically reduced. An attractive way out is offered by fusible interlayer which can effectively remove oxide films. In the course of bonding, a fusible interlayer is melted, most of the molten metal is expelled from the joint by the pressing load, and the composition of the remaining portion approaches that of the parent metal owing to diffusion processes. This type of interlayer can be used to join both similar and dissimilar metals.

To assure the requisite actual contact between the mating surfaces, the metal at the interface must first undergo a certain amount of plastic deformation. With fusible interlayers, the pressing load may be applied as soon as the interlayer is melted, following which the bonding pressure can be brought down to a value just sufficient to hold the pieces in place. Fusible interlayers also make it possible to use lower bonding pressure as they prevent the formation of microdiscontinuities in the joint.

In diffusion (pressure) brazing the interlayer solidifies isothermally. In diffusion bonding, the pressing load expels the interlayer from the joint, and a characteristic diffusion bond is formed at once within individual areas. These areas grow in size with an increase in bonding pressure until all of the liquid phase is expelled from the joint. The use of fusible interlayers subjected to pressing is widely practised both in and outside the Soviet Union, and the procedure is carried out in vacuum. By varying the bonding conditions or simply the size and shape of the mating surfaces it is possible to make a joint typical of diffusion bonding or with individual areas typical of diffusion brazing.

Additionally, fusible interlayers serve to activate the mating surfaces. The liquid phase promotes the detachment, dispersal and dissolution of oxide films. The activating effect of the interlayer is accentuated still more, if it contains small amounts of elements capable of reducing or converting the oxides to readily fusible compounds. These elements may be carbon, boron, and alkaline elements. During the first step of bond formation, the liquid interlayer wets the parent metal and breaks the bonds between the atoms of the parent metal and of the chemisorbed or physically adsorbed material. Thus, in addition to the activation by heat and deformation typical of diffusion bonding, now use is also made of the activation by the liquid phase.

Importantly, the use of a liquid interlayer makes less stringent the requirements for the mechanical preparation of the mating surfaces. By wetting the parent metal, the interlayer can dissolve it or smoothen asperities. This is where the analogy with diffusion brazing ends. In diffusion brazing, the luquid phase fills the gap between the mating surfaces, then it solidifies and makes a brazed joint. In diffusion bonding with a fusible interlayer the latter is no longer needed at the end of the first step and is expelled from the joint. It would be ideal if it could be removed completely, because the interlayer is usually inferior to the parent metal in mechanical properties. Dispersal of the interlayer is promoted by the associated diffusion processes; the thinner the interlayer, the faster it will be dispersed. The dispersal time for the interlayer can be found by solving Fick's diffusion equation for a varying diffusion source in one dimension. If at initial time t the concentration of the diffusing element in the interlayer h thick is $C = C(x) = C_0$ and the amount of diffusing element remains unchanged, $Q = C_0 h$, then the concentration of the diffusing element is distributed in accord with the following equation:

$$C = \frac{C_0 h}{2\sqrt{\pi D t}} \exp(-x^2/4Dt) \tag{10-11}$$

Obviously, the concentration of the element will be a maximum at the centre where $x = 0$, and equal to

$$C = C_0 h / 2\sqrt{\pi D t} \tag{10-12}$$

On setting the maximum tolerable concentration of the element at the centre of the interlayer, we can find the time required for this concentration to be achieved as a function of the initial thickness of the interlayer:

$$t = C_0^2 h^2 / 4\pi D C^2 \tag{10-13}$$

It is seen from Eq. (10-13) that the dispersal time of the interlayer to the specified concentration of the element is proportional to the square of the initial concentration and thickness. Therefore, in order to enhance the chemical homogeneity of the joint, the largest possible proportion of the interlayer material must be expelled from the joint.

From the foregoing it may be concluded that a fusible interlayer should meet the following requirements.

(1) It should activate the mating surfaces.

(2) It should be easy to expel from the joint when the bonding pressure is applied to the assembly.

(3) In composition, the interlayer should match as closely as possible the parent metal so as to ensure high chemical homogeneity in the joint.

(4) It should be able to diffuse quickly into the parent metal without causing embrittlement.

(5) The interlayer should melt at a temperature which is below that of undesirable structural transformations in the parent metal and lies in a range where the parent metal has high plasticity.

From phase diagrams for binary systems it is seen that the fusible interlayers intended for use in joining high-temperature alloys can be based on nickel systems such as Ni-Mn, Ni-Si, Ni-B, Ni-Pd, Ni-Ti, Ni-Zr, Ni-Nb, and Ni-Be. These systems may contain small amounts of other elements capable of activating the process and of depressing the melting point of the system. In selecting these alloying elements, it is essential to consider their effect on the high-temperature behaviour of the parent alloy.

In the choice of fusible interlayers and bonding variables, it is important to consider not only the strength characteristics of the resultant joint, but also its susceptibility to embrittlement. Embrittlement may be caused by the formation of boride, silicide, carbide and intermetallic interlayers. Whereas one should expect the formation of borides, silicides and carbides if the corresponding elements are present in the parent metal, the formation of intermetallic compounds is far more difficult to predict. In high-temperature alloys, the principal and most offending intermetallic compound is the sigma-phase. The formation of the sigma-phase can be predicted

on the basis of the procedure proposed by Sims for the design of high-temperature alloys.

The primary objective in the choice of an interlayer and bonding variables is to assure a maximum homogeneity in the joint. The presence of soft or hard interlayers in the finished joint has an adverse effect on its performance. We have already considered the effect of soft interlayers. A hard interlayer lying across the force flux and outside of the stress concentration zone will not usually affect the strength of the joint. In the presence of stress concentrators, the strength of the joint sharply declines, because the stronger materials have a higher sensitivity towards stress concentrators. If a hard interlayer lies across the force flux, it will quickly lose its plasticity. In doing so, it will break down and turn into a stress concentrator, and this will have a negative effect on the behaviour of the joint.

A further factor that can affect the formation of a diffusion bond is the manner in which fusible or dispersable interlayers are made or used. Fusible interlayers may be introduced in the joint in the form of foil or powder, deposited by chemical, electroplating, carbonyl, explosion, hot, diffusion or some other processes, or by gas-flame, electric-arc or plasma spraying, cathode sputtering, vacuum evaporation, etc.

To date, several types of fusible and easy-to-expel interlayers have been investigated, notably those involving Ni-Mn, Ni-Si and Ni-B systems which form the basis for most commercial brazing alloys. In the Soviet Union, the most commonly used brazing alloys are ВПр-7, ВПр-11, ВПр-11-40Н, and some others. The composition (in %) of ВПр-7 is Ni-33Mn-Nb. That of ВПр-11 is Ni-15Cr-4.5Si-2.5B-0.5C-4Fe. An interlayer can best be expelled when the wetting angle does not exceed 5-8°, but the parent metal dissolves only slightly.

The effect of the pressing load on the bond formation in ЭП99 alloy with a fusible interlayer is demonstrated in the plot of Fig. 10-9. As the pressing load is raised to 15 MPa, the thickness of the interlayer rapidly decreases until it reaches a thickness of 10-20 μm at a holding time of 6 min. This thickness results from the expulsion of the interlayer and diffusion processes. Joint strength with such interlayers depends on their composition and structure which are in turn determined by dissolution and diffusion processes. In most cases, when joints are made at a

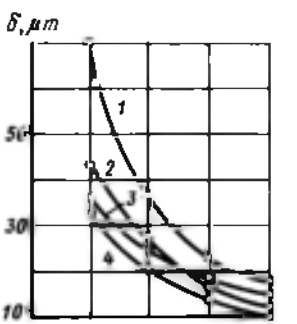

FIG. 10-9 Effect of pressing load on the thickness of the interlayer in ЭП 99 alloy
1, 2 — ВПр-7; *3, 4* — ВПр-11; *1, 4* — 1 448 K; *3* — 1 423 K; *2* — 1 473 K. Holding time, 6 min

pressing load of 10-15 MPa and an appropriate temperature the interface is hard to detect by microscopic examination. For example, in a joint made with a ВПр-7 interlayer the metal in the bond zone consists of solid-solution grains; after ageing, the γ'-phase precipitates. As follows from local X-ray diffractometry, the composition of the interlayer does not remain the same across its thickness. The distribution of the interlayer elements obeys the equation of diffusion from a source with a limited quantity of material. From studies into the effect of bonding temperature on the thickness and composition of interlayers it has been learned that raising the temperature to 1 473 K promotes the expulsion of the interlayer from the joint. The maximum concentration of manganese in the centre of the interlayer at a temperature of 1 473 K and a pressing load of 10 MPa is 5.4% (Fig. 10-10). In joints

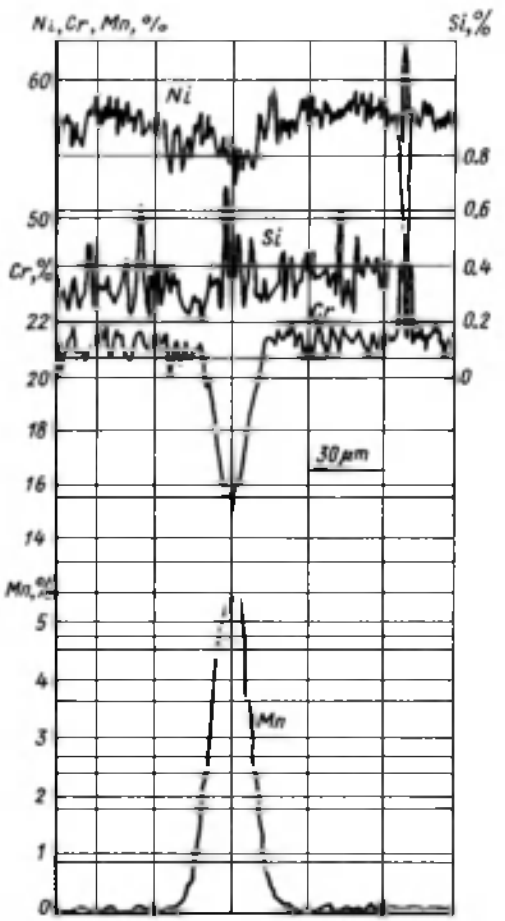

FIG. 10-10 Distribution of elements in joints of ЭП 99 alloy with ВПр-7 interlayer (T = 1 373 K, p_b = 10 MPa, t = 6 min)

with a ВПр-11 interlayer the composition of the metal in the bond zone is likewise very close to that of the parent metal (Fig. 10-11), but, with the temperature brought down to 1 398 K, the joint may include high-melting-point borides.

A study has been undertaken into the use of fusible interlayers from binary Ni-Mn and Ni-Si systems and evaporated manganese and silicon. It has been found that

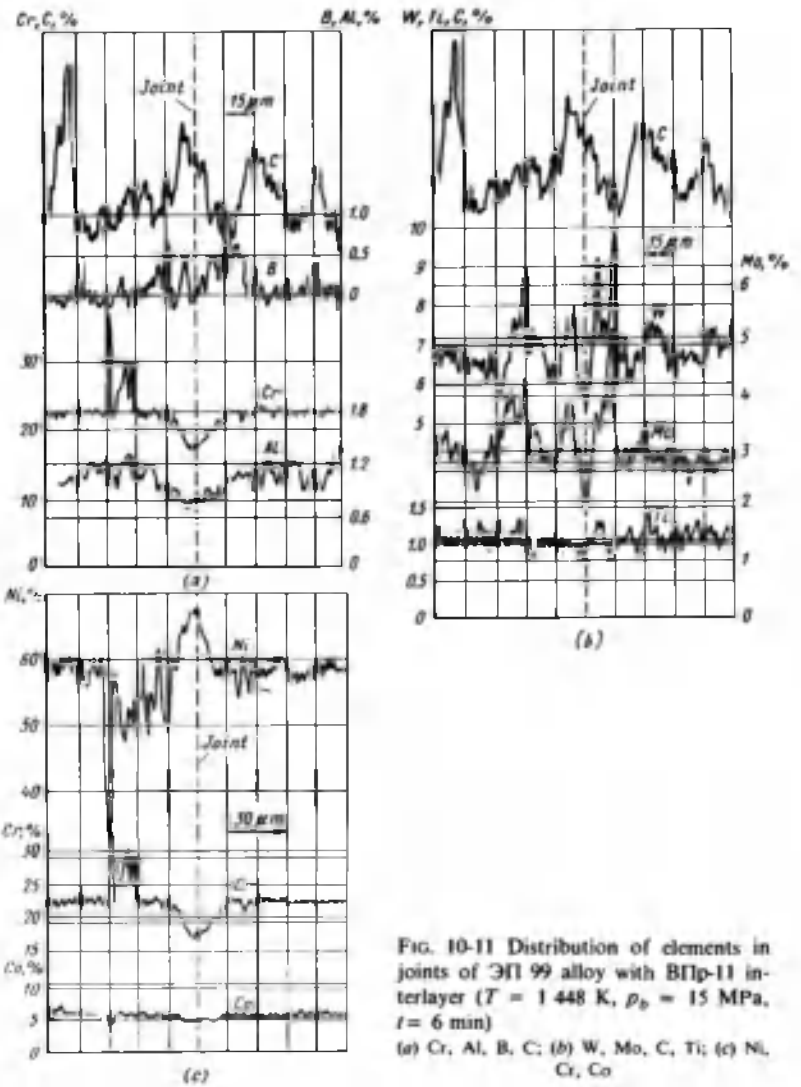

FIG. 10-11 Distribution of elements in joints of ЭП 99 alloy with ВПр-11 interlayer (T = 1 448 K, p_b = 15 MPa, t = 6 min)
(*a*) Cr, Al, B, C; (*b*) W, Mo, C, Ti; (*c*) Ni, Cr, Co

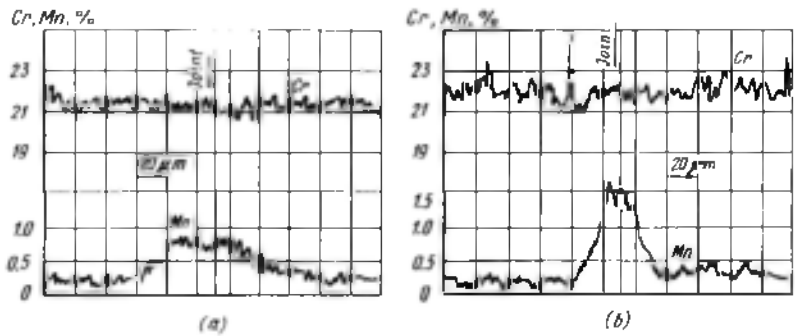

FIG. 10-12 Distribution of chromium and manganese in joints of ЭП 99 alloy with ВПр-7 interlayer
(*a*) after diffusion treatment for 16 h at 1 373 K; (*b*) after diffusion treatment for 300 h at 1 173 K

owing to expulsion and the subsequent dissolution and diffusion processes the composition of the metal in the bond zone is close to that when joints are made with ВПр-7 and ВПр-11 interlayers. The mechanical properties have been found to be very much alike, too.

In service, components from high-temperature alloys are heated to 1 173 K or even higher. Investigations have shown that in service the chemical homogeneity of diffusion bonds improves (Fig. 10-12), the concentration of chromium being practically fully and that of magnesium substantially homogenized. To evaluate the effect of bonding variables on the performance of joints, mechanical tests were made on lap- and butt-joined cylindrical specimens on a gas-dynamic test bench and in actual service conditions. The objectives of the tests were to determine the short- and long-time strength of the joints, their fatigue strength and thermal endurance. The short-time strength of lap joints in ЭП99 alloy was found to depend but little on the bonding conditions. For example, in the case of joints made with a ВПр-7 interlayer at a temperature in the range 1 428-1 473 K and at a pressing load in the range 5-15 MPa, the shear strength as measured at 1 173 K was 66-102 MPa. With any set of bonding conditions the spread in values was considerable, and the results were consistent only when the joints were made at 1 473 K. Somewhat better results were obtained in the diffusion bonding of ЭП455А alloy: when joints were made at a temperature of 1 473 K and a bonding pressure of 15 MPa the shear strength rose to 101-129 MPa as against 74-84 MPa in joints made at a bonding pressure of 1 MPa. The increase in the bonding pressure to 15 MPa in making joints in ЭП99 alloy with a ВПр-11 interlayer at a temperature of 1 398 K had no marked effect, and the shear strength as measured at 1 173 K was in the range 73-100 MPa. When the bonding temperature was raised to 1423-1448 K, the shear strength of the joints increased to 89-131 MPa. In the case of lap joints in ЭП99 and ЭП648 alloys with an evaporated manganese interlayer 10 μm thick, made at a temperature of 1 173 K, a bonding pressure of 15 MPa and a holding time of 6 min, the short-time strength as

measured at 1 423 K was 81-97 MPa. In the case of joints made with a silicon interlayer at the same temperature, bonding pressure and holding time, the strength was 137 MPa.

A comparison of joints made in ЭП99 alloy with different interlayers shows that the ВПр-11 interlayer provides a better strength than the ВПр-7 interlayer does. The strength of joints made with an evaporated manganese interlayer is close to that obtained with a ВПр-7 interlayer. Silicon interlayers, too, produce high mechanical properties, but sometimes cracking occurs and the joints fail at room temperature because of the brittle silicides present in the joint.

The effect of bonding temperature and pressure on the quality of diffusion-bonded joints is more pronounced when tests are run for a longer time. The time to failure for lap joints made in ЭП99 alloy with a ВПр-11 interlayer trebles when the bonding pressure is raised from 5 to 15 MPa and is 1400-2000 h as measured at 1 173 K. The rise in bonding temperature from 1348 to 1398 K has a far more pronounced effect. The increase in both the bonding temperature and the bonding pressure improves the strength of joints and makes them more consistent in quality. From an analysis of the high-temperature strength of joints, it has been found optimal to make joints at a temperature of 1 473 K with a ВПр-7 interlayer and at a temperature of 1 423-1 448 K with a ВПр-11 interlayer. For either material, the optimal bonding pressure is in the range 10-15 MPa, the holding time is 6 min, and the vacuum is 1.3×10^{-2} Pa. For joints made with a ВПр-11 interlayer and under optimal conditions the long-time strength is about three times the figure for joints made with a ВПр-7 interlayer. Their thermal endurance is likewise high. It appears that of all binary systems, the Ni-Si system is most promising.

The bonding conditions given above have also been used to make joints between dissimilar high-temperature alloys. The butt joints thus made matched in strength the weaker of the two metals.

In his study, Stolyarov [13] used a Cr-Ni-Pd alloy interlayer to make joints in ХН65ВМТЮ alloy. Tests were made on cylindrical specimens 65 mm long and 22 mm in diameter, pressure-brazed according to the procedure developed by a team under Puzrin at the Paton Electric Welding Institute. The properties of specimens subjected to simple stabilization after bonding at 1 073 K (for 12 h) were very low, notably their ductility. When the specimens were subjected to homogenizing annealing in the form of multistage ageing (for 4 h at 1 273 K, then for 8 h at 1 173 K, and finally for 15 h at 1 123 K), the properties of the joints were improved considerably. In fact, as tested at 1 023 K they matched the parent metal in mechanical properties. This improvement after homogenizing annealing is due to the dispersal of the interlayer and its hardening by disperse phases at the expense of the parent metal.

In regard to nickel-base alloys, an important indicator of the high-temperature behaviour is their long-time strength defined as the maximum constant stress that will not cause failure during a specified time interval at a specified temperature. For stationary turbines, long-time strength is especially important because they have to be operated continuously (such as in gas-turbine generating units) for as long as up to 30 000 h. The tests of joints in ХН65ВМТЮ alloy at 1 023 K show that when they

are made with a Cr-Ni-Pd interlayer (at T = 1493-1500 K, and t = 20 min) and subjected to homogenizing annealing (for 4 h at 1 273 K, then for 8 h at 1 173 K, and finally for 15 h at 1 123 K), the joints have a long-time strength of 137 MPa over 30 000 h. As in short-time tests, specimens undergo brittle failure at the interlayer (the percent elongation is 0.43-2.66%). The long-time strength of the parent metal is 176 MPa. The long-time percent elongation is 2.3-11%.

In his work, Khimushin [14] used boron-carrying fusible interlayers to join Ni-base and Co-base high-temperature alloys to each other and to various combinations of other alloys. Tests for static oxidation at 1 473 K in air for 100 h showed that the bond zone was not oxidized. Both at room and elevated temperature, the joints matched the parent metal in strength. In making joints, low bonding pressures around 0.3-0.5 MPa were used. In contrast, the holding time was extended to 24 h at a temperature of 1 444 K. In the circumstances, the brazing alloy solidified

TABLE 10.1

Material	Bonding temperature, K	Bonding pressure, MPa	Holding time, min	Bonding atmosphere
Nickel	1273	15	10	Vacuum 1.3×10^{-2} Pa, or hydrogen with a dew point of 313 K
Monel	1173	15	10	
ХН80ТБЮА	1473	20	6	Vacuum $1.3 \times \times 10^{-2}$ Pa
ХН65ВМТЮ	1148	20	20	Same
ЖС6К, ЖС6КП	1273	20	10	Same
ХН75МБТЮ	1423-1448	20-30	6	Same
ЭП99	1423-1448	35-40	6	Same
Udimet 700 (with Ni-Co interlayer)	1463	83	4 h	Same
TD-Ni (with Co-alloy interlayer)	1366	135	2 h	Same
TD-Ni-Cr (two-stage procedure)	978	206	1 h	Vacuum $2.6 \times \times 10^{-3}$ Pa
	1463	15	2 h	Same
ХН77ТЮ (with evaporated carbon interlayer)	1323-1373	30	5	Vacuum $1.3 \times \times 10^{-2}$ Pa
ХН75МБТЮ + ЭП99 (with ВПр-7 or evaporated manganese interlayer)	1473	10-15	6	Same
ХН77МБТЮ + ЭП99 (with ВПр-11 interlayer)	1423-1448	10-15	6	Same
Nimonic 90 (with nickel foil interlayer)	1373	32	15	Same

isothermally. This procedure is less effective than that in which, instead of solidifying isothermally, the greater proportion of the interlayer is expelled from the joint. Removal of the interlayer by pressing permits the metal in the bond zone to achieve a very high homogeneity within a few minutes after bonding. The subsequent heat treatment, however short, homogenizes the chemical composition of the bond metal practically completely.

Suggested conditions for the diffusion bonding of nickel and some of nickel-base alloys, with fusible and easy-to-expel interlayers, with solid interlayers, and without interlayers are listed in Table 10-1.

The quality of diffusion-bonded joints can be tested with any one of several methods. The most promising method is ultrasonic flaw detection (for turbine components the shadow variety of the method is used). A good deal can be achieved through active control of bonding conditions.

10.5 Bonding of Magnet Alloys

As their name implies, these alloys go to make magnets. Because the alloys are difficult to machine and have a high hardness and brittleness, it is customary to make built-up magnets. Another reason for the use of built-up magnets is that it is not always possible to assure directional solidification throughout the bulk of a large and odd-shaped casting, so important for good performance. The easiest way to make built-up magnets is by means of diffusion bonding which has a number of advantages to offer in this application.

Materials for permanent magnets are classed according to some leading engineering property related to their chemical composition, or according to the structural state yielding the highest coercive force H_c. A high coercive force is the most important property for good magnets.

For economic reasons and also because they possess a happy combination of magnetic properties, the magnet alloys most commonly used commercially are those based on the Fe-Ni-Al system. A most outstanding example is Magnico alloys. Their coercive force ranges between 3.98×10^5 and 6.4×10^5 A m^{-1} at a residual magnetic induction $B_r = 1.0$-1.35 T. Owing to a highly convex demagnetization curve, their specific energy, W, is as high as 30 kJ m^{-3}.

According to Bidermann and Kneller, precipitation-hardening magnet alloys may be classed into three groups:

(1) Alloys in which a second, less magnetic or nonmagnetic phase is formed in a ferromagnetic matrix (the precipitate of iron carbide or iron tungstate in iron).

(2) Alloys which decompose owing to concentrational disproportionation into structurally equivalent components one of which has either definitely ferromagnetic or nonmagnetic properties. Neither can be identified as the matrix or the precipitate (Fe-Ni-Al, Ni-Cu, and Co-Ni-Cu alloys).

(3) Alloys in which fine, more ferromagnetic particles are embedded in a weakly magnetic or nonmagnetic matrix (copper alloys containing a few percent iron).

It should be noted that there is no clearly defined boundary between the three groups of magnet alloys. A feature common to those in the second group is that

they appear homogeneous in a microscope. Their constituents are finely dispersed and the degree of dispersion materially affects the coercive force.

A distinction of Fe-Ni-Al alloys is that in all cases an increase in magnetic energy is achieved through an increase in coercive force H_c. When B_r is very high, both coercive force and magnetic energy are relatively small. On the other hand, commercial applications need magnet alloys which have both a high residual induction and a high magnetic energy. This need is filled by a more recent addition, the Fe-Ni-Co-Al alloy. In this alloy, a maximum coercive force is achieved at the very onset of homogeneous precipitation when the precipitating particles are too small to be detected in a microscope.

In Magnico alloys, coercive force is determined by the degree of dispersion of the decomposition products of solid solution, β and β_2, their chemical composition, and the structure of the disperse particles. It can be raised by substantial internal stresses. A maximum of magnetic energy is provided by a special heat treatment. Both hardened and annealed, Magnico alloy has a microstructure similar to Ni-Al. The dark phase in the annealed specimen is the β-phase embedded in the matrix high in Ni-Al.

Most frequently, cast permanent magnets based on the Fe-Ni-Co-Al system are used. They have high magnetic properties and stable magnetic characteristics.

The chemical composition and magnetic characteristics of some of the Soviet-made magnetic alloys are given in Tables 10-2 and 10-3.

PROCEDURE FOR THE DIFFUSION BONDING OF MAGNET ALLOYS. In designing a procedure for the diffusion bonding of magnet alloys the optimal bonding variables

TABLE 10-2

Alloy	Chemical composition, %					
	Ni	Al	Co	Cu	Fe	Others
ЮН14ДК24	13.5-14.5	7.5-8.5	23.5-24.5	2.5-3.5	Balance	—
ЮН14ДК25БА	13.5-14.0	8.0-8.5	24.0-26.0	3.5-4.0	Balance	Nb, 0.8-1.0; Ti, 0.3 min
ЮНДК35Т5БА	14.0-14.5	6.8-7.2	34.5-35.5	3.3-3.7	Balance	Ti, 4.7-4.5

TABLE 10-3

Alloy	B_r, T	H_c, A m^{-1}	BH, kJ m^{-3}
ЮН14ДК24	1.25	4.8×10^4	36
ЮН14ДК25БА	1.3	5.7×10^4	56
ЮНДК35Т5БА	1.38	81.12×10^3	72

The effect of temperature on the magnetic properties of ЮН14ДК24 alloy when bonded with a powdered nickel interlayer is illustrated in Fig. 10-13. As is seen, already at 823-873 K the coercive force was reduced by as much as 5-7%. The subsequent two-stage tempering (for 2 h at 893 K and for 8 h at 853 K) completely restored the initial value of H_c of the specimens bonded at 923-1023 K. When bonding was done at a higher temperature, the subsequent tempering could only partly restore the values of H_c and B_r. In the case of specimens bonded at a temperature above the Curie point (1 098 K), two-step tempering restored the magnetic characteristics to values 50% higher than those which existed immediately after bonding.

Figure 10-14 illustrates the effect of temperature on the magnetic properties of ЮН14ДК25БА alloy of high coercive force. The data were obtained on bonded specimens which were subjected to two-step tempering for 2 h at 893 K and for 8 h at 853 K, or for 2 h at 863 K and for 11 h at 833 K. In the case of specimens bonded at 973-1 023 K, this form of postheat-treatment restored their magnetic properties to within acceptable limits (H_c within 5% and B_r within 4%).

Alloy ЮНДК35Т5БА containing 5% Ti has a fine-grained structure, is brittle, and possesses low strength. Even in the "as-delivered" condition it has cracks, and is not practically suitable for diffusion bonding. The effect of temperature on the coercive force of this alloy, when tempered after bonding for 20 h at 833 K, is illustrated in Fig. 10-15. As is seen, H_c is reduced by as much as 15-20% already at a bonding temperature of 923 K. The subsequent tempering restores the original value of H_c.

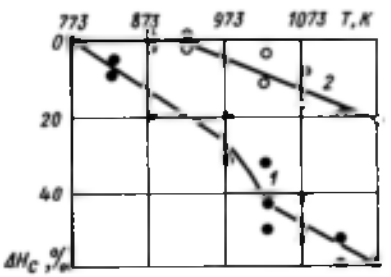

FIG. 10-13 Effect of bonding temperature on the magnetic properties of joints in ЮН14ДК24 magnet alloy + ЭАА steel (p_b = 9.8 MPa, t = 5 min; powdered nickel interlayer)
1 — as bonded; *2* — as postheat-treated (tempering)

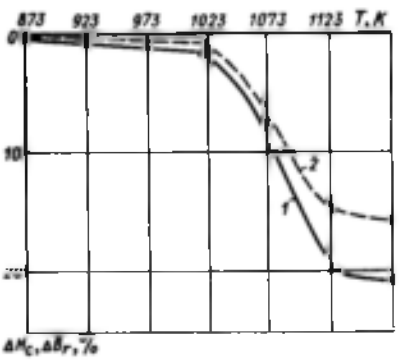

FIG. 10-14 Effect of bonding temperature on the magnetic properties of joints in ЮН14ДК25БА magnet alloy + ЭАА steel
1 — as bonded; *2* — as postheat-treated (tempering)

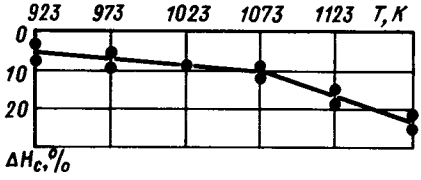

FIG. 10-15 Effect of bonding temperature on the magnetic properties of joints in ЮНДК35Т5БА magnet alloy + ЭАА steel in as-bonded condition and after postheat-treatment (tempering). (p_b = 19.6 MPa, t = 10 min, powdered nickel interlayer)

The basic consideration in choosing the bonding variables for magnetic alloys is to preserve the magnetic characteristic in the weldment. This calls for the measurement of the demagnetization curve of the material before and after welding on a ballistic machine. From the curve thus found, the values of B_r and H_c are calculated. The coercive force of magnets is measured by means of a solenoid, with its circuit open. The field intensity in the gap of a magnet system can be measured with a Hall transducer or with a calibrated test coil connected to a ballistic galvanometer.

Bibliography

1. BAKSHI, O. A., SHRON, R. Z. *The Ultimate Tensile Strength of Joints Bonded with a Soft Interlayer.* "Svarochnoye proizvodstvo", 1962, No. 5, pp. 6-10 (in Russian).
2. KAZAKOV, N. F. *Diffusion Bonding of Materials.* Mashinostroyeniye, Moscow, 1976 (in Russian).
3. KAZAKOV, N. F., ABRAMOV, V. V., MASHKOVA, N. A. *The Study and Design of Procedures for the Diffusion Bonding of Magnetically Hard to Magnetically Soft Materials with a Powdered Interlayer.* In: "Vacuum Diffusion Bonding of Metals, Alloys and Nonmetals", Part 1. PNILDSV, Moscow, 1968, pp. 107-112 (in Russian).
4. KAZAKOV, N. F., GERBERG, A. N., MASHKOVA, N. A. *Vacuum Diffusion Bonding of Magnet Alloys.* "Svarochnoye proizvodstvo", 1967, No. 12, pp. 35-36 (in Russian).
5. KARAKOZOV, E. S. *Solid-State Joining of Metals.* Metallurgiya, Moscow, 1976 (in Russian).
6. KVASNITSKY, V. F., KOKH, B. A., SAFONOV, A. I. *Vacuum Diffusion Bonding of High-Temperature Alloys.* Znaniye, Leningrad, 1969 (in Russian).
7. KVASNITSKY, V. F., KULIK, S. G., ZLAMAN, N. G. *A Study into the High-Temperature Deformation of High-Temperature Alloys in Vacuum Diffusion Bonding.* "Transactions of the Nikolayev Shipbuilding Institute". 1972, iss. 53, pp.37-41 (in Russian).
8. MALEVSKY, Yu. B., MARKASHOVA, L. I., PEREVERZEV, D. D. *Some Aspects of Bulk Processes in the Diffusion Bonding of Nickel.* In: "Vacuum Diffusion Bonding of Metals, Alloys and Nonmetals". The 7th All-Union Conference. PNILDSV, Moscow, 1972, pp.17-18 (in Russian).
9. MEDOVAR, B. I. *Welding of High-Temperature Austenitic Steels and Alloys.* Mashinostroyeniye, Moscow, 1966 (in Russian).

10. MIKHAILOV-MIKHEYEV, P. B. *Handbook of Metals for Turbines and Engines.* Mashgniz, Moscow-Leningrad, 1961 (in Russian).
11. MOVCHAN, B. A. *A Study into the Physical Parameters of High-Temperature Creep and Long-Term Failure of Some High-Temperature Alloys under Heavy Stresses.* "Fizika metallov i metallovedeniye", 1967, Vol. 24, No. 6, pp. 1082-1091 (in Russian).
12. MUSIN, R. A., ANTSIFEROV, V. N., KVASNITSKY, V. F. *Diffusion Bonding of High-Temperature Alloys.* Metallurgiya, Moscow, 1979 (in Russian).
13. STOLYAROV, V. N. *High-Temperature Performance of Joints Made in Nickel Alloys by Diffusion Bonding and Pressure Brazing.* "Avtomaticheskaya svarka", 1971, No.1, pp. 9-11 (in Russian).
14. KHIMUSHIN, F. F. *High-Temperature Steels and Alloys.* Metallurgiya, Moscow, 1969 (in Russian).
15. DAVIES, R. J., STEPHENSON, N. *Diffusion Bonding and Pressure Brazing of Nimonic 90 Nickel-Chromium-Cobalt Alloy.* "British Welding Journal", 1962, No.3, pp. 139-148.
16. DUVALL, D. S., OWCZARSKI, W. A., PAULONIS, D. F. *TLP-Bonding: a New Method for Joining Heat-Resistant Alloys.* "Welding Journal", 1974, 53, No.4, pp. 205-214.
17. MOORE, T. J., HOLKO, K. H. *Solid-State Welding of TD-Nikel Bar.* "Welding Journal", 1970, No.9, pp. 395S-409S.

11

Bonding of Cemented Carbides and of a Cemented Carbide to Steel

V.S. Samoilov

11.1 Bonding of Cemented Carbides

The need to join a cemented carbide to another exists in tool-making and machine-building in the fabrication of components whose size or shape preclude the use of powder metallurgy techniques. Typical examples are blanks for the cylinders and pistons of high-pressure compressors, components for compression moulds with intricate shape of cavities, etc.

A cemented carbide material (a carbide alloy) consists of a carbide phase (WC, TiC, or TaC) dispersed in a matrix or binder (Co, Ni, or Mo). In its preparation, a mixture of the respective carbide and binder is sintered at a temperature sufficient to produce a liquid phase.

Diffusion bonding is widely used to join die and mould components made of WC + Co carbides. The composition and physico-mechanical properties of some Soviet-made WC-Co (or tungsten) carbides are given in Table 11-1.

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11. MOVCHAN, B. A. *A Study into the Physical Parameters of High-Temperature Creep and Long-Term Failure of Some High-Temperature Alloys under Heavy Stresses.* "Fizika metallov i metallovedeniye", 1967, Vol. 24, No. 6, pp. 1082-1091 (in Russian).
12. MUSIN, R. A., ANTSIFEROV, V. N., KVASNITSKY, V. F. *Diffusion Bonding of High-Temperature Alloys.* Metallurgiya, Moscow, 1979 (in Russian).
13. STOLYAROV, V. N. *High-Temperature Performance of Joints Made in Nickel Alloys by Diffusion Bonding and Pressure Brazing.* "Avtomaticheskaya svarka", 1971, No.1, pp. 9-11 (in Russian).
14. KHIMUSHIN, F. F. *High-Temperature Steels and Alloys.* Metallurgiya, Moscow, 1969 (in Russian).
15. DAVIES, R. J., STEPHENSON, N. *Diffusion Bonding and Pressure Brazing of Nimonic 90 Nickel-Chromium-Cobalt Alloy.* "British Welding Journal", 1962, No.3, pp. 139-148.
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Diffusion bonding is widely used to join die and mould components made of WC + Co carbides. The composition and physico-mechanical properties of some Soviet-made WC-Co (or tungsten) carbides are given in Table 11-1.

TABLE 11-1

Designation	Composition, wt %		Density, ρ, $\times 10^2$ kg m^{-3}	Ultimate bending strength, σ_b, $\times 10^7$ Pa	Hardness, Rockwell A	TCLE, α, $\times 10^{-6}$ K^{-1}
	WC	Co				
BK6M	94	6	15.0	135	90	4.9-5.0
BK6	94	6	14.8	145	88.5	4.9-5.0
BK8	92	8	14.6	160	87.5	5.0-5.1
BK10	90	10	14.4	160	87.0	5.3
BK15	85	15	14.0	180	86.0	6.0-6.3
BK20	80	20	13.5	190	84.5	6.5

In recent years, the scarcity of tungsten [5] has spurred the use of titanium carbide or carbonitride dispersed in a Ni-Mo matrix and of titanium carbide dispersed in an iron-base matrix alloyed with chromium and nickel. The chemical composition and some properties of the former are given in Table 11-2, and those of the latter in Table 11-3.

In contrast to iron-base steels and alloys, cemented carbides have a high ultimate compressive strength and a low ductility (the limit of deformation in compression is 0.5-5%), and this dictates a special approach to their bonding. The decisive factor is surface quality, notably out-of-parallelism and roughness. Because

TABLE 11-2

Designation	Composition, wt %				Density, ρ, $\times 10^3$ kg m^{-3}	Ultimate bending strength, σ_b, $\times 10^7$ Pa	Ultimate compressive strength, σ_c, $\times 10^7$ Pa	Hardness, Rockwell A	TCLE, α, $\times 10^{-6}$ K^{-1}
	Ti(CN)	TiC	Ni	Mo					
TH-20	—	79	15	6	5.6	100	350	89.5	7.0
KHT-16	74	—	19.5	6.5	5.8	110	390	89.0	8.8

TABLE 11-3

Designation	Composition, wt %				Density, ρ, $\times 10^3$ kg m^{-3}	Ultimate bending strength, σ_b, $\times 10^7$ Pa	Ultimate compressive strength, σ_c, $\times 10^7$ Pa	Hardness Rockwell A
	TiC	Fe	Cr	Ni				
TC30XH	70	21.9	5.4	2.7	5.8	115-125	450	89-90
TC40XH	60	29.2	7.2	3.6	5.9	125-140	410	87-88

of this cemented carbides are joined to one another with a nickel interlayer. With it, the mating surfaces conform more readily to each other's contour, and the rate of diffusion processes is boosted owing to the high diffusion activity of nickel. High-cobalt cemented carbides (such as BK15 and BK20) can be bonded without an interlayer.

In the course of bonding, the binder (Co, Ni, Fe) interdiffuses with the interlayer material to form a joint. Since Ni, Co and Fe have a high mutual solubility, no brittle intermetallic compounds are formed in the bond zone.

The carbide phase cannot diffuse directly. In some cases, it dissolves in the interlayer material, so that the nickel is saturated with carbon and with the metal of the carbide. The solubility is a maximum for tungsten and iron. In this case, the bond zone is a series of solid solutions of cobalt or iron in nickel with an addition of the carbide-forming metal, and has a microhardness which is only a few units below that of the workpieces. It is, however, only a few micrometres wide, so it does not affect the performance of the bonded joint.

Cemented carbides can be bonded in any type of machine in which the vacuum is no less than 2.66×10^{-3} Pa and the bonding temperature is at least 1 373 K. Use can also be made of a shielding atmosphere (hydrogen, argon) and of heating by a glow discharge [4]. Prior to bonding, the mating surfaces must be ground and lapped so that they are flat to within 0.01-0.02 mm. Lapping should preferably be done immediately before bonding. Then the components should be thoroughly degreased with acetone and rubbed with a cotton swab moistened with alcohol. The recommended bonding conditions, interlayer materials and thicknesses are given in Table 11-4.

Vacuum diffusion bonding can also be used to repair cemented-carbide parts (dies and the like) with large chipped areas. In such cases, the interlayer is deposited by electroplating or by evaporation in vacuum. The plot of joint strength as a function of evaporated film thickness in Fig. 11-1 shows that the strength is a maximum when the deposit is 4×10^{-7}-8×10^{-7} m thick [3].

TABLE 11-4

Pair to be bonded	Interlayer material	Thickness, mm	Bonding conditions		
			T, K	p_b, MPa	t, min
BK6 + BK6	Ni	0.005	1373-1423	14.7-17.6	18-20
BK8 + BK8	None		1373-1473	13.7-15.7	
BK15 + BK15				12.7-14.7	
BK20 + BK20				11.7-13.7	
TH20 + TH20	Ni	0.005	1373-1423		25-30
TC30XH + TC30XH					
TC40XH + TC40XH		0.01	1323-1373	9.8-11.7	

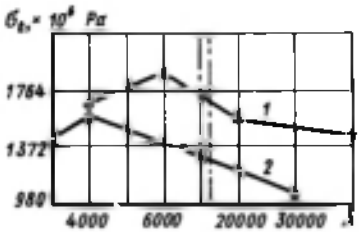

FIG. 11-1 Strength of BK + BK carbide joints as a function of thickness of plating
1 — cobalt; *2* — nickel

11.2 Bonding of Cemented Carbides to Steel

Because of scarcity and low ultimate tensile and bending strength, the cemented carbides used in tool making are mostly shaped tips or inserts joined in one way or another to a steel tool blank. Therefore, the performance of carbide-tipped tools depends to a great extent on the quality of the joint. The process best suited to this application is diffusion bonding in vacuum [4, 7]. Because of a substantial difference in thermal expansion, thermal conductivity and ultimate compressive strength, bonding is done with an interlayer, of which the best grades are H2 nickel and Permalloy (50НП). Most frequently, the interlayer is a piece of foil cut to the contour of the pieces being joined [4]. In several cases, good results have been obtained with an interlayer of porous iron [7]. Joint strength varies with the thickness of the interlayer; the maximum strength has been obtained with a nickel interlayer 0.08-0.10 mm thick. Joint strength is also dependent on the quality of the mating surfaces, notably roughness and parallelism of the abutting faces. Optimal results have been obtained with a roughness of 0.80 μm and an out-of-parallelism of 0.02 mm (Fig. 11-2). The bonding conditions, too, affect the quality of joints. This is especially true of bonding temperature (Fig.11.3), whereas bonding pressure and duration of pressure have a less noticeable effect. The optimal conditions for joining BK cemented carbide to steel are T = 1323-1373 K, p_b = 11.7-14.7 MPa, and

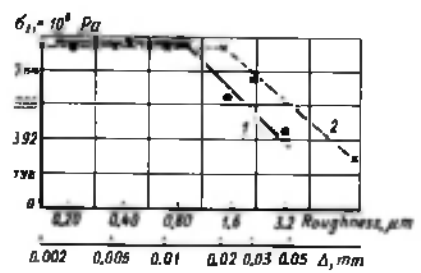

FIG. 11-2 Tensile strength of BK20 carbide + steel joints as a function of (*1*) surface roughness and (*2*) parallelism of end faces

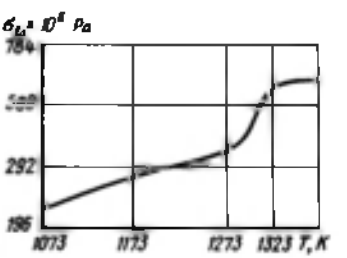

FIG. 11-3 Tensile strength of BK20 carbide + 18Х2Н4ВА steel joints as a function of bonding temperature (p_b = 9.8 MPa, t = 10 min)

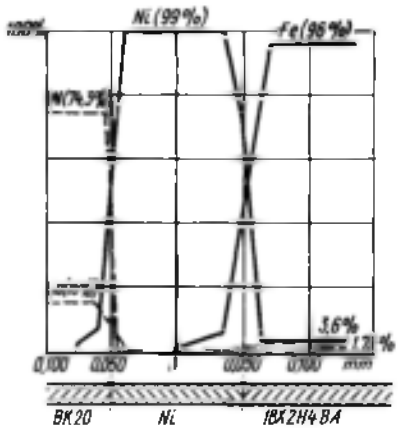

FIG. 11-4 Distribution of elements in a BK20 carbide + Ni + 18X2H4BA steel joint (diffusion-bonded at T = 1 323 K, p_b = = 9.8 MPa, and t = 1.5 min). Element percentages are laid off as abscissae

t = 10-12 min. When made under optimal conditions, joints between BK cemented carbide and steel have an ultimate shear strength of 392-450 MPa, an ultimate twist strength of 274-294 MPa, and an ultimate tensile strength of 588-882 MPa.

When a cemented carbide is diffusion-bonded with a nickel or Permalloy foil interlayer, their interdiffusion produces a transition zone (Fig. 11-4) in which practically all of the elements of the metals being joined are present. The element most active in the formation of the transition zone is nickel which, given optimal bonding conditions (T = 1323 K, p_b = 9.8-1.9 MPa, and t = 10-12 min), diffuses into the cemented carbide to a depth of 25-30 μm. In doing so, it replaces the cobalt of the binder, whereas WC grains partly dissolve in the nickel to form a Ni-Co solid solution. Thus, at the interface with the interlayer the binder is a solid solution of nickel, tungsten and cobalt, in which the Ni and W content decreases on moving away from the interface between the nickel interlayer and cemented carbide. When the duration of pressure is over 10-12 min, WC grains eagerly dissolve in the Ni-Co solid solution. In the process, the WC grains are refined and rounded (Fig. 11-5). The replacement of the cobalt in the binder by nickel and the partial dissolution of WC grains lower the microhardness of the solid solution in the transition zone by 1×10^3-1.2×10^3 MPa as compared with the original value. The diffusion of nickel into the steel mainly proceeds along grain boundaries. For bonding under optimal conditions, the depth of the diffusion zone is 20-30 μm along grain boundaries and 8-10 μm in the bulk. Owing to the diffusion of nickel into the steel, the microhardness of the steel in the zone adjacent to the interlayer goes up by 4×10^2-5×10^2 MPa as compared with the original value. The iron of the steel diffuses into the interlayer to a depth of 45-55 μm. So, if the interlayer is 0.005-0.02 mm thick, some iron can pass through the interlayer into the cemented carbide. The diffusion of iron reduces the ductility of the interlayer, because of which the relaxation capacity of the interlayer is decreased and the internal stresses are increased. This is why the interlayer between a cemented carbide and a steel must be no less than 30 μm thick.

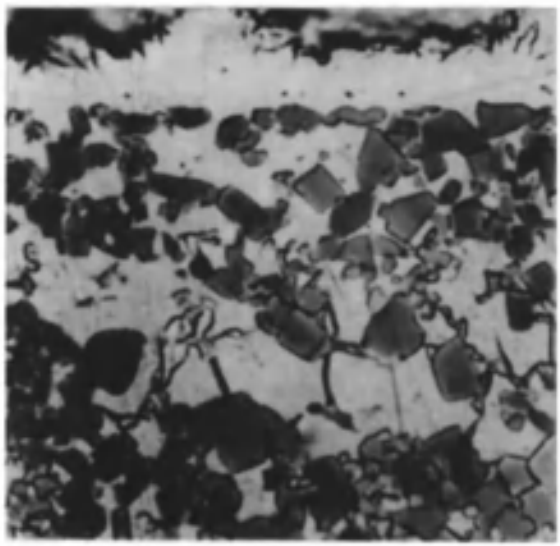

FIG. 11-5 Microstructure of the transition region in BK20 carbide + Ni + 18X2H4BA steel joint diffusion bonded at $T = 1\,373$ K, $p_b = 9.8$ MPa and $t = 30$ min. × 1350

Since cemented carbides and steels differ in thermal expansion, diffusion bonding in vacuum gives rise to internal stresses of the first kind, which produce distortion in the weldment and may cause failure of the joint after bonding or in service. The magnitude and character of internal stresses depend on the properties of the materials involved and the bonding technique and procedure used. Therefore, in designing the latter, every effort must be made to minimize the internal stresses to a point where they cannot affect the performance of the weldment.

Tensile stresses are especially dangerous for cemented carbides. Generated by joining on the surface of the cemented carbide and, as a consequence, at the cutting tip of the tool, tensile stresses can combine with working stresses, their sum can exceed the safe limit, and the carbide tip will chip out or crack. From experience in the fabrication of carbide tools by brazing [5] it is known that the stressed state of the cemented carbide in a brazed tool can radically be changed by using martensitic steels (such as 18X2H4BA, 5XHB, 5XHT, and 7XГ2BM) for tool shanks or backers.

As has been learned from a study into the tensile stresses occurring in the joining of BK20 cemented carbide to Ст 3 steel and 18X2H4BA martensitic steel with nickel and Permalloy interlayers 0.02 mm, 0.04 mm and 0.10 mm thick, diffusion bonding in vacuum produces substantially lower internal stresses than brazing. Brazing a cemented carbide to Ст 3 steel produces a tensile stress of 486 MPa at the surface of the cemented carbide and a compressive stress of 775 MPa in the bond

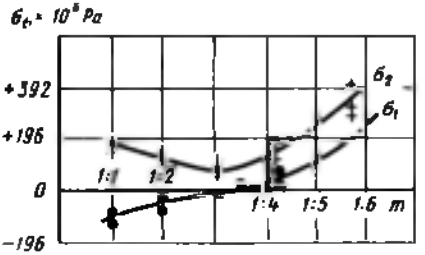

FIG. 11-6 Internal residual stresses in the cemented carbide alloy of a BK20 carbide + Ni + 18X2H4BA steel joint as a function of the carbide-to-steel thickness ratio: σ_1, at the surface of the carbide alloy; σ_2, in the bond zone

zone. In the case of diffusion bonding the respective figures are 16.5 MPa and 406 MPa. The level of stresses decreases with increasing thickness of the interlayer. The situation is reversed in joining BK20 cemented carbide to 18X2H4BA steel — now compressive stresses are generated at the surface of the cemented carbide and tensile stresses in the bond zone. The level of stresses goes down as the thickness of the interlayer is increased. A further factor affecting the magnitude of internal stresses is the relative thickness of the cemented carbide and the steel (the ratio of their thicknesses, m). The stresses are at their lowest when $m = 1:3$ (Fig. 11-6). This is why it is well advised to use martensitic steels for the tool shanks that are to be carbide-tipped by vacuum diffusion bonding.

After diffusion treatment at constant temperature, the weldment should be allowed to cool in the chamber to 1 133 K, then at a higher rate in air. In the course of high-rate cooling, the steel backer is partly hardened (to a hardness of 38-50 Rockwell C). As a result of the martensitic transformation of the steel, the steel backer increases in volume, so compressive stresses are generated at the surface of the cemented carbide tip, and negligible tensile stresses in the bond zone. The difference in stresses in the case of martensitic steels is far smaller than it is in the case of ferritic and pearlitic steels. Since practical diffusion-bonded articles are of complex shape, the formation of stress concentrators is inevitable. However, the reduced difference in stress across the section of the cemented carbide leads to a reduced stress at points of concentration. In cases where the steel backer cannot be given a hardness of over 40 Rockwell C, it will be a good plan to treat the article at a low temperature (343 K) for an extended period of time.

Diffusion bonding is especially advantageous in the fabrication of tools operating under alternating loads (such as blanking dies, impact-extrusion punches, etc.). Table 11-5 summarizes the results of tests involving carbide-faced impact-extrusion punches (punch height, 30 mm; impact force, 230-250 MPa; impact rate, 50 min^{-1}).

As is seen from Table 11-5, most durable punches are made when diffusion bonded according to the recommended procedure. The backer hardness of 30-40 Rockwell C is sufficient for most practical applications in blanking dies, spinnerets, and compression moulds. For tools operating under high loads (such as forging dies), however, the hardness must be raised 54-60 Rockwell C.

TABLE 11-5

Materials	Interlayer thickness, mm	Backer hardness, Rockwell C	Bonding conditions T, K	p_b, MPa	t, min	Impacts to failure, thou	Failure
Ст45 + Cu + ВК20 (brazing)	0.10	28-30	1323		10	Under 10	Breakage at carbide
						360	Chipping in carbide
18Х2Н4ВА + Ni + + ВК20	0.04	39-41					
		38-40		9.8		480	Same
18Х2Н4ВА + Ni + + ВК20		38-41	1223 950			5-10	Breakage at bond
	0.10	39-41			5	70	Same
		40-42	1323	4.9		260	Same
18Х2Н4ВА + Permalloy + ВК20		38-41		9.8	10	400	Major chipping in carbide

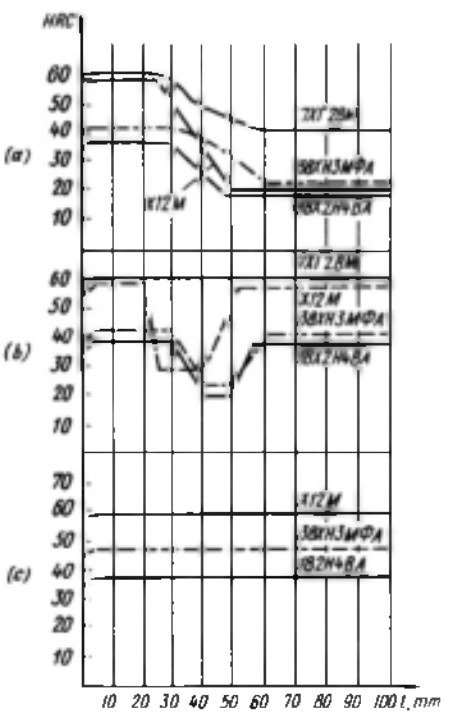

FIG. 11-7 Hardness of the steel part of a bonded joint obtained with different bonding procedures

(*a*) bonding and heat treatment from single heating in the vacuum chamber; (*b*) bulk hardening followed by bonding; (*c*) bonding followed by bulk hardening. Distance to the bond zone is laid off as abscissae

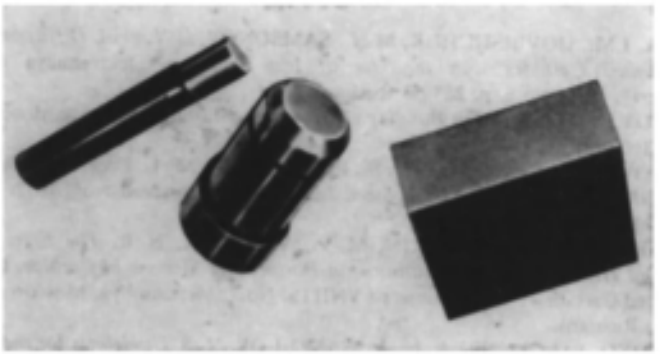

FIG. 11-8 Several parts fabricated by vacuum diffusion bonding

TABLE 11-6

Step	Equipment	Temperature, K	Duration, min
Preheating	Furnace	623	20-25
Reheating	Same	1023-1123	30-40
Heating for hardening	Salt bath	1323-1343	13-15
Hardening at constant temperature	Same	573-673	3-4
Rinsing	Water bath	333-353	2-4
Drawing	Furnace	473-523	60-90

In long articles, a transition zone with a hardness of 30-32 Rockwell C is formed within 20-30 mm of the bond zone in the course of bonding and the subsequent heat treatment from the bonding temperature (Fig. 11-7). When subjected to a heavy load, the steel tool backer might deform at such a transition zone beyond the limits of serviceability. To avoid this, a uniform hardness must be produced over the entire length of the steel backer by means of postweld hardening. The hardening temperature should be specified according to the grade of steel used, and the hardening time according to the size of the article. The recommended hardening variables for parts 63-65 mm in diameter and 40-50 mm long are listed in Table 11-6.

Examples of tools (forging dies, blanking dies, compression moulds for powder metallurgy) are shown in Fig. 11-8.

Bibliography

1. GALINSKY, E.R., SARANDI, R.L., KAZAKOV, N.F. *Equipment and Procedures for the Fabrication of Bimetallic Die Components by Diffusion Bonding*. In: "Vacuum Diffusion Bonding". PNILDSV, Moscow, 1973. Iss. 5, pp. 107-109 (in Russian).

2. MUKHA, I.M., DOVBISHCHUK, M.N., SAMSONOV, G.V. *et al. Diffusion Bonding of Cemented Carbides with the Use of Ion Heating.* "Elektronnaya obrabotka materialov", 1971, No.6, p. 283 (In Russian).
3. KAZAKOV, N. F. *Diffusion Bonding of Materials.* Mashinostroyeniye,Moscow, 1976, pp. 312-360 (In Russian).
4. KAZAKOV, N. F., SAMOILOV, V. S., POLYAKOVA, M. L. *Vacuum Diffusion Bonding of BK20 Cemented Carbide to Steel.* "Svarochnoye proizvodstvo", 1972, No.2, pp. 18-19 (In Russian).
5. KLOCHKO, N. A., MAKHOTKIN, M. V., MOINOVA, N. R. *The Effect of Steel Grade and Heat Treatment Conditions on Postbrazing Stresses in Carbide Tools.* In: "Cemented Carbides". Transactions of VNIITS, No.7. Metallurgiya, Moscow, 1967, pp. 51-59 (in Russian).
6. KLYACHKO, L. I., KUDRYA, N. A., SAMOILOV, V. S. *Guidelines for the Economy of Tungsten in Cemented Carbides.* Tsvetmetinformatsiya, Moscow, 1976, pp. 6-7, 16-21 (in Russian).
7. MUKHA, I. M., DOVBISHCHUK, M. N., KALNENKO, Ye. I. *The Use of Porous Interlayers in the Diffusion Bonding of Cemented Carbides to Steel.* "Poroshkovaya metallurgiya", 1972, No. 10, pp. 101-105 (In Russian).
8. POLYAKOVA, M. L., POPOV, V. G., SAMOILOV, V. S. *The Fabrication of Carbide-Tipped Impact Extrusion Dies.* "Kuznechnoshtampovochnoye proizvodstvo", 1977, No.2, pp. 46-47 (in Russian).
9. SARANDI, R. L., KARKHIN, V. A. *Stress Distribution in a Carbide Insert Diffusion-Bonded to a Steel Shank.* "Elektrotekhnicheskaya promyshlennost", 1976, No.4, pp. 4-6 (in Russian).
10. TUMANOV, V.I. *The Properties of WC-Co Alloys.* Metallurgiya, Moscow, 1971, pp. 7-13 (in Russian).

12

Repair and Reconditioning by Diffusion Bonding

N.V. Filatov and *G.V. Zakorin*

12.1 General

Frequently machine parts break well before they have served their useful life. It is, therefore, economical to bring them back to service by repair or reconditioning. This may have as its objective solely to restore the shape, physical and mechanical properties of a part, or to "rejuvenate" it completely as if it had come from its original maker.

In this field, diffusion bonding has many advantages to offer. Among other things, the diffusion bond has the same physical and mechanical properties as the

2. MUKHA, I.M., DOVBISHCHUK, M.N., SAMSONOV, G.V. *et al. Diffusion Bonding of Cemented Carbides with the Use of Ion Heating.* "Elektronnaya obrabotka materialov", 1971, No.6, p. 283 (In Russian).
3. KAZAKOV, N. F. *Diffusion Bonding of Materials.* Mashinostroyeniye,Moscow, 1976, pp. 312-360 (In Russian).
4. KAZAKOV, N. F., SAMOILOV, V. S., POLYAKOVA, M. L. *Vacuum Diffusion Bonding of BK20 Cemented Carbide to Steel.* "Svarochnoye proizvodstvo", 1972, No.2, pp. 18-19 (In Russian).
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6. KLYACHKO, L. I., KUDRYA, N. A., SAMOILOV, V. S. *Guidelines for the Economy of Tungsten in Cemented Carbides.* Tsvetmetinformatsiya, Moscow, 1976, pp. 6-7, 16-21 (in Russian).
7. MUKHA, I. M., DOVBISHCHUK, M. N., KALNENKO, Ye. I. *The Use of Porous Interlayers in the Diffusion Bonding of Cemented Carbides to Steel.* "Poroshkovaya metallurgiya", 1972, No. 10, pp. 101-105 (In Russian).
8. POLYAKOVA, M. L., POPOV, V. G., SAMOILOV, V. S. *The Fabrication of Carbide-Tipped Impact Extrusion Dies.* "Kuznechnoshtampovochnoye proizvodstvo", 1977, No.2, pp. 46-47 (in Russian).
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12.1 General

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In this field, diffusion bonding has many advantages to offer. Among other things, the diffusion bond has the same physical and mechanical properties as the

remainder of the part; reconditioning leaves unaffected the critical elements; the techniques and procedures are equally applicable to cast iron, nonferrous metals and alloys; the cost of reconditioning is a fraction of that of making a new part of the same quality.

Vacuum diffusion bonding can be used to repair or recondition an article by replacing its worn or broken portions, by building up worn surfaces, by adding compensators, or otherwise utilizing the structural reserves of the component. Accordingly, it is customary in the Soviet Union to class all components to be repaired or reconditioned into three groups, namely critical frictional parts (CFP), critical nonfrictional parts (CNP), and noncritical parts (NP).

Basic requirements for reconditioned components in the CFP group are as follows. (1) Misalignment between the portions being joined must be within the tolerance permitting normal operation of the reconditioned part with the mating component. (2) After repair or reconditioning, the dimensions must lie within the limits specified for the next repair. (3) There should be no distortion of the geometry within frictional areas. (4) The strength of the joint must be 2 or 3 times the operating load.

Requirements for reconditioned components in the CNP group are the same as those for components in the CFP group, except item (3). Requirements for reconditioned components in the NP group only include items (2) and (4).

12.2 Reconditioning by Replacement of a Defective Portion

The defective (worn) portion of a component is cut off, a new one is made, and is joined instead of the removed portion. A high-quality joint can only be produced by diffusion bonding.

RECONDITIONING OF COMPONENTS IN THE NP GROUP. After they are loaded in the vacuum chamber, the parts are mated together by directly putting them together "face-to-face". Proper alignment or location can be secured with a suitable jig or fixture, or by applying a light pressure. As an alternative, the parts to be joined can be "tacked" to each other with studs, dowels, keys, and similar devices (Fig. 12-1). The sides of the joint should have a surface quality comparable with that given by finish turning or milling. The requirements for surface degreasing are the same as in all applications of vacuum diffusion bonding.

RECONDITIONING OF COMPONENTS IN THE CFP GROUP. These are mostly rotating parts with surfaces machined to a high degree of finish, such as gears, spindles,

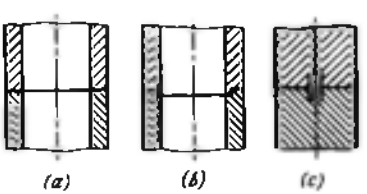


FIG. 12-1 Methods of mating parts in the NP group
(*a*) direct "face-to-face"; (*b*) by means of shoulders; (*c*) by means of locating pin

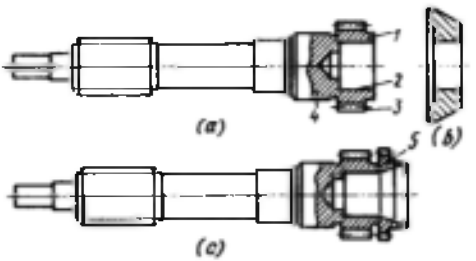


FIG. 12-2 Reconditioning of an automotive transmission primary shaft by replacement of the synchronizer ring gear

(*a*) primary shaft after cutting off the worn portion; (*b*) blank; (*c*) reconditioned shaft; *1* — centring surfac *2* — needle bearing journal; *3* — helical gear; *4* — ball-bearing journal; *5* — position of the bond zone in the recon ditioned component

shafts, axles, etc. In them, the bond zone must lie away from frictional surfaces. The worn portion of a component can be replaced with a piece which is (1) a blank with ample allowance for subsequent machining or (2) a fully finished element calling for no subsequent machining. The choice depends on the capabilities of the repair department or the repairability of the component.

Case (1). The preparatory steps are very simple, because the mating portions are held in sufficient alignment by shoulders. The locating shoulder on the blank is given an ample allowance for subsequent machining after diffusion bonding. An example is the reconditioning of an automotive transmission primary shaft in which the synchronizer ring gear is to be replaced (Fig. 12-2). The operation includes the following steps:

— the worn part is drawn (tempered) by h.f. induction heating;

— the worn ring gear is cut off and the surface is prepared for bonding (Fig. 12-2 *a*);

— a blank is fabricated (Fig. 12-2*b*);

— the blank is joined to the shaft by diffusion bonding;

— the reconditioned component is machined to drawing (this includes turning, gear-cutting, etc.), so that an allowance is left for the subsequent grinding of the synchronizer taper end;

— the machined component is given a heat treatment and its quality is tested;

— the synchronizer taper end is ground;

— the diffusion bond is tested for quality.

The datum surfaces used in cutting off the worn ring gear are the journal, *4*, for the ball-bearing in the gearbox casing (see Fig. 12-2) and the face of the helical gear, *3*. This is done in order to preserve the working surfaces, that is, the helical gear and the journal for the needle bearing, *2* (in this way, the bond plane is distant from them). The bonding pressure is applied in the right direction with the aid of a ball fixture such as shown in Fig. 12-3. After reconditioning, the shaft is given a standard heat treatment. Preferably, it should be conducted in a medium which can prevent the reoxidation of the machined surfaces (this may be a bath of molten salt or a

FIG. 12-3 Fixture securing proper position of the primary shaft under the compression rod of a bonding machine
1 — shaft to be reconditioned; *2* — blank; *3* — thrust disc; *4* — ball

suitable coating). The bond quality can be checked by a twist test under a load which is three times the service value. Because diffusion bonding produces consistently sound joints it is sufficient to test one shaft of a lot.

Case (2). Since the mating portions are fully machined, it is essential to align them properly prior to bonding and to check their dimensions. Locating surfaces on the mating portions are machined with the use of suitable arbours, with reference to the datum surfaces on the complete component. For example, in the case of an internal cylindrical datum surface use may be made of an expansion arbour (Fig. 12-4). In the case of an external cylindrical datum surface (say, the ring gear), it is advisable to employ a split bushing (Fig. 12-5). The portions are mated to a

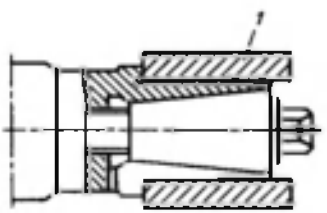


FIG. 12-4 Expansion arbour used to centre the pieces when centring is done relative to internal locating (datum) surfaces: *1* — component

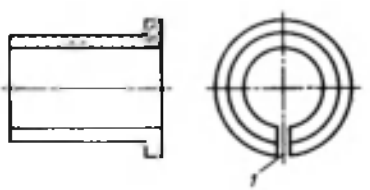


FIG. 12-5 Split bushing used to centre the pieces when centring is done relative to an external locating (datum) surface: *1* — split

TABLE 12-1

Dia. of centring surface, mm	Tolerance, μm		Length of locating surface, mm
	shaft	hole	
3-6	+9	+13	2-3
	+1	0	
6-10	+12	+16	2-3
	+2	0	
10-18	+14	+19	2-3
	+2	0	
18-30	+12	+13	3-5
	+2	0	
30-50	+14	+15	3-5
	+2	0	
50-80	+16	+18	5-7
	+3	0	
80-120	+19	+21	5-7
	+3	0	

force fit, so they are aligned accurate to within 10 μm. This is well within the limits specified for the assembly in which the reconditioned component will be used. The tolerances on the diameter and length of locating shoulders recommended for use in the reconditioning of components in the CFP and CNP groups are listed in Table 12-1.

The mating surfaces in diffusion bonding must be at right angles to the direction of the pressing load.

In reconditioned components it is important to preserve the dimensions of the working surfaces within specified limits. This involves resort to various schemes and devices preparatory to and during the bonding operation.

Above all, the bond zone should be located as far away from the working surfaces as practicable. If this cannot be done, a way out is to machine what are called free deformation zones which permit the near-bond metal to undergo deformation without affecting the abjacent material. An example is shown in Fig. 12-6. As is seen, a free deformation zone is prepared by removing stock in the near-bond zone on either (a) the component being reconditioned or (b) the replacement part. For the joint to match the parent metal in strength, the dimensions of a free deformation zone may be h = 0.5-1 mm and b = 6-10 mm. The outside diameter at the bond zone is brought to the desired value either by subsequent machining or by removing some stock prior to joining, with allowance for the likely plastic deformation of the material at and near the joint in the course of bonding. Removal of some stock is

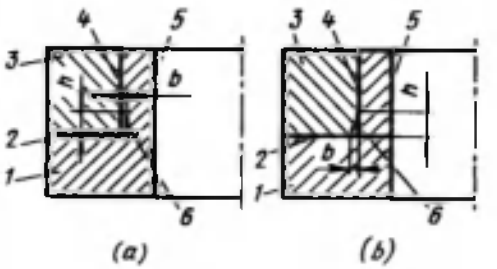


FIG. 12-6 Manner of joint preparation in the reconditioning of parts in the CFP group
1 — component to be reconditioned; *2* — diffusion-bond plane; *3* — replacement part; *4* — locating (centring) surface; *5* — primary locating surface of the component; *6* — free deformation zone

shown in Fig. 12-7, where *h* and *b* have the same values as given earlier. The plastic deformation of the asperities left by machining and of the stock next to the joint causes the parts to be joined to move axially in the direction of the applied bonding pressure. The amount of upset is anywhere between 0.35 mm and 0.60 mm. It should be taken into account in preparing the component for reconditioning so as to preserve the desired geometry and relative position of the component elements. This can be done when cutting off the worn portion of the component or making a replacement for it.

Jigs and fixtures are important factors in the repair and reconditioning of components by diffusion bonding. Importantly, they should uniformly distribute the

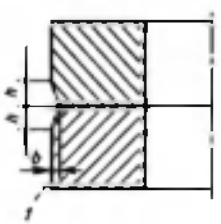


FIG. 12-7 Recess in the near-bond zone made to allow for the bulk plastic deformation in the course of bonding:
1 — primary locating surface

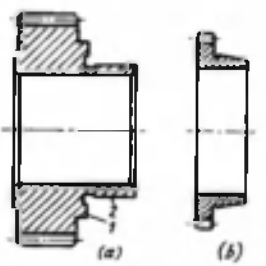


FIG. 12-8 Preparation for the reconditioning of a double-ring transmission gear by replacement of the worn smaller ring gear
(*a*) serviceable part of the compound gear; (*b*) replacement for the worn ring gear; *1* — bond surface; *2* — centring (locating) surface

FIG. 12-9 Installation of a wear-compensator in a ball-bearing housing

applied bonding pressure over the joint area . In order to meet this requirement the axis of the assembly should be aligned with the direction of the applied pressure and the latter must be at right angles to the mating surfaces. Heating must be sufficient, but it should not cause the melting of the mating surfaces.

An example of reconditioning in which a worn portion of an assembly is replaced by a fully machined one is that involving the double ring gear of an automotive transmission. This operation consists of the following steps:

— the worn portion (the straight-tooth ring gear) is drawn (tempered) by h.f. induction heating;

— the worn portion is cut off and the surface is prepared for bonding (Fig. 12-8*a*);

— a new ring gear is made for replacement (Fig. 12-8*b*);

— heat treatment is applied and its quality is tested;

— the taper portion of the synchronizer is ground;

— after the ring gear has been thus reconditioned, a new bushing is installed.

RECONDITIONING OF COMPONENTS IN THE CNP GROUP. These include bearing housings, valve seats and other critical components in which nonfrictional surfaces are usually worn. Most often, they are reconditioned by installing wear compensators (Fig. 12-9). It is important that compensators should have a high hardness. In most cases, however, they cannot be hardened by heat treatment after they have been installed in the reconditioned part. Therefore, it is usual to make compensators from a strong material, to hard-face them by spraying, and to press-fit or cement them in place. Where this form of anchoring is not sufficiently reliable, it is preferable to secure a compensator in place by diffusion bonding.

12.3 Repair Through the Use of Structural Reserve of the Component

Sometimes, a component has to be rejected because it has worn out of true on one side or locally, whereas its strength, dimensions, shape, etc. have remained intact. This is especially true of gears. Obviously, such components can be restored to service by utilizing their reserves, and they will have the same service life as they had prior to rejection. An example is an automotive reverse compound gear (Fig. 12-10). Such gears are usually rejected because of a substantial wear on the face of the smaller gear, whereas the teeth of the two gears show but an insignificant reduction in thickness. Therefore, the compound gear as a whole can be returned to service by turning the smaller gear back to front and joining it by diffusion bonding. This can be done in any one of two ways.

Case (1). Two rejected compound gears are available. On one, the smaller ring gear is machined so as to leave enough stock for joining the similar ring gear removed from the other compound gear (Fig. 12-10*a*). Since two rejected components are utilized, the service life of the reconditioned assembly is extended by 50%. All the tolerances and fits involved should be taken as suggested in Table 12-1.

Case (2). The smaller ring gear is cut off, turned back to front and joined to the same compound gear. In order to preserve the dimensions, an annular insert should

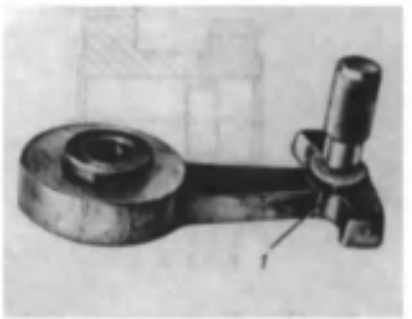


FIG. 12-12 Machine-tool lever reconditioned by diffusion bonding: *1* — bonded joint

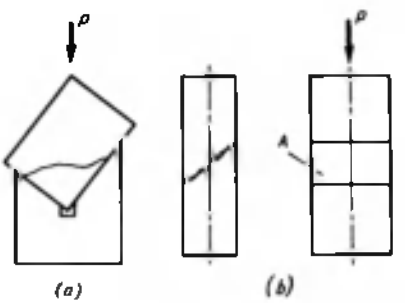


FIG. 12-13 Various techniques for the repair of parts in the case of an oblique fracture
(*a*) with a prism; (*b*) with a compensator, *A*

mating surfaces could be positioned at right angles to the applied bonding pressure (Fig. 12-13*a*). In the case of an oddly shaped fracture, the failed portion must be cut out and a compensator installed (Fig. 12-13*b*).

12.5 Industrial Use of Vacuum Diffusion Bonding for Repair and Reconditioning

In the Soviet Union, vacuum diffusion bonding has successfully been used for repair and reconditioning by automobile fleet operators, farms and factories. A variety of techniques and procedures have been developed for a range of components some of which are shown in Fig. 12-14. A further example (at *1* in

FIG. 12-14 Parts reconditioned by diffusion bonding

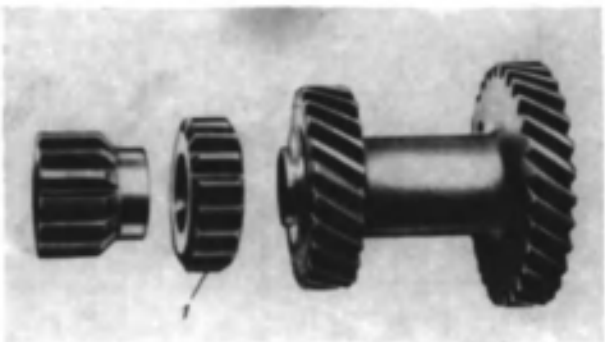
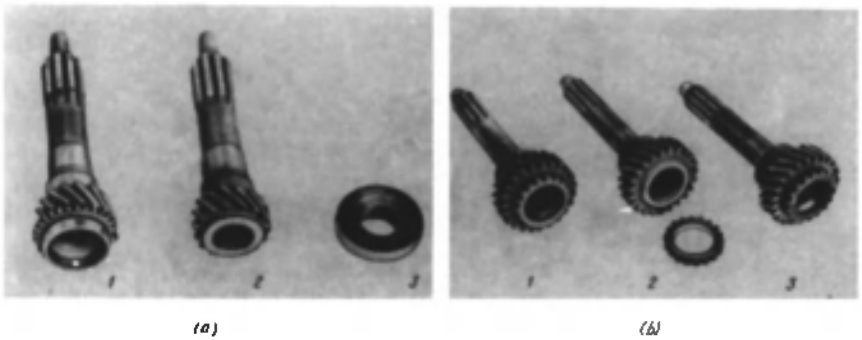


FIG. 12-15 Components of an automotive countershaft compound gear: *1* — gear replaced at the time of repair

Fig. 12-15) is the middle straight-tooth gear of the compound gear on an automotive transmission countershaft. This gear is usually the first to fail by the time of overhaul. Its replacement with a new one by means of diffusion bonding makes the entire assembly acceptable for further service. In quality, it compares with a new one, and the cost of reconditioning is by 63% less than that of a new compound gear. Figure 12-16 shows one of the five reclaimed bushings for a gear-type pump, all fabricated from a single worn bronze bushing. The rejected bushing was sliced into five discs, these were joined each to a steel backer, and a perforated bronze foil was inserted inside. The bushing thus made is comparable in quality with a new all-bronze one, but its cost is one-fourth and it takes one-fifth the original amount of bronze. Figure 12-17 illustrates the reconditioning of the primary-shaft ring gears for the transmission gearboxes of GAZ and ZIL trucks. The cost of reconditioning is 30% the cost of a new component in the case of a GAZ truck and 39% the cost of a new component in the case of a ZIL truck.

FIG. 12-16 Bushing with its bearing surfaces bronze-lined by diffusion bonding



(a) (b)

FIG. 12-17 Reconditioning of primary shafts
(*a*) GAZ truck: *1* — shaft with a worn synchronizer ring gear, *2* — worn ring gear removed, *3* — blank for manufacture of a new synchronizer ring gear after bonding; (*b*) ZIL truck: *1* — shaft with a worn ring gear, *2* — worn ring gear removed and a new one is prepared for bonding, *3* — shaft after a new ring gear has been joined by diffusion bonding

In the case of blanking dies, the carbide plates often come off the steel backer because of a poor brazed joint. Diffusion bonding completely does away with the failure and adds a good deal to the service life of the tools.

As we have seen, it does pay to use diffusion bonding for the purpose of repair and reconditioning as it substantially extends the service life of the components, cuts back on the use of new parts, and saves metal.

13

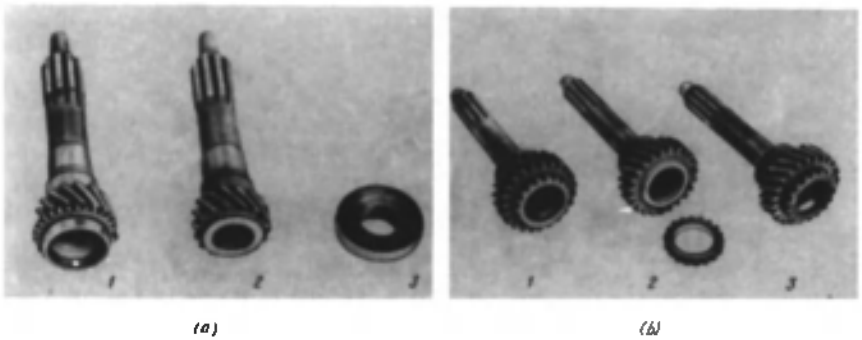
Bonding of Porous Materials

E. K. Soluyanova and *N. F. Kazakov*

13.1 Porous Cermet Filter Media and Their Uses

Porous cermet filter media are widely used as fine filters in the fuel, oil and hydraulic systems of aircraft, tractors, Diesel locomotives, compressors and many other machines. They can advantageously be used to remove products of wear and corrosion, dust, sand, scale particles and other particulate matter from air and liquid streams.

In regard to fluids, porous cermets provide their fine filtering action because their pores form circuitous paths. Their permeability increases with increasing size



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In regard to fluids, porous cermets provide their fine filtering action because their pores form circuitous paths. Their permeability increases with increasing size

and number of open pores, retention time, and pressure drop across the filter. It decreases with increasing thickness of the filter medium, viscosity of the filtered fluid, and friction between the fluid and the filter element [1].

Porous cermets offer a number of advantages over filters using fabric, felt, pressboard, ceramics, porcelain, gauzes and some other organic materials. They are sturdier, can operate over a wide temperature range, and possess a controlled porosity and good permeability. They stand up well to sharp changes in temperature, lend themselves readily to machining and welding, and are easy to regenerate.

The good filtering capacity of porous cermets has made them attractive in gas-liquid analysis apparatus, gas-dispensing systems, oil-moisture separators, catalysts, flame barriers, and elsewhere. Some sintered porous materials have shown a good performance as seals and packings in farm machinery, turbines, pipe and boiler fittings and accessories. The fabrication of seal rings by powder metallurgy techniques simplifies the manufacturing operation, cuts down the material lost at the machining step, and avoids the use of the more expensive and, sometimes, scarce materials, such as corrosion-resistant steels, brass, and bronze.

Porous cermet elements are widely used in hand-operated pneumatic machines, reinforcing-bar welding units and concrete casting yards for noise abatement. As tests have shown, the use of a porous cermet noise muffler can bring down the noise level by 5 to 25 dB, depending on the sound pressure level, without any loss in the power output of the associated machinery [5].

Porous cermets can be fabricated from carbon steels, a variety of corrosion-resistant steels, nickel, Monel metal, copper, bronze, titanium, tungsten, molybdenum and some other materials.

Industrially, use is most commonly made of cermet filter elements fabricated from powdered carbon steels, corrosion-resistant steels, nickel, titanium, aluminium, and bronze. The chemical composition of some of the powders along with selected properties are given in Table 13-1.

The dimensions and shapes of the most commonly used cermet filter elements are shown in Table 13-2.

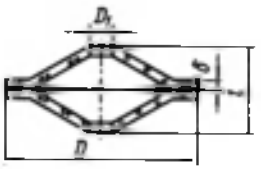
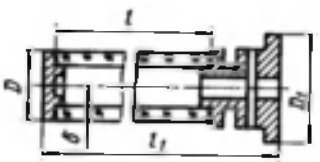
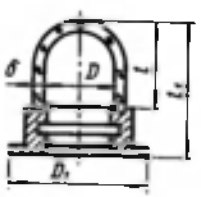
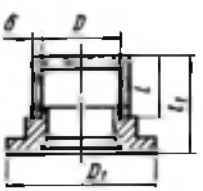
The most commonly used shapes are bushings, discs, rings, and tapering sleeves. Their performance is substantially improved when a porous cermet component is joined to a solid-metal backer. Long porous cermet tubes (1 m long and more) with a uniformly distributed porosity (about 30-70%) are difficult to fabricate, as this calls for the use of sophisticated and expensive equipment. Also, it is sometimes impossible to make long tubes having an elaborate configuration. The usual practice is to make them by joining together shorter pieces. Joining can be done by several processes, depending on the material involved and the service conditions expected [7]. Most frequently this is done by welding. Unfortunately, the conventional welding processes do not produce consistently sound joints. Often, failure occurs because of a poor contact between the mating surfaces due to the presence of oxides in the weld zone. Short of failure, their presence impairs the joint strength and the filtering performance of the product.

Quality joints in porous materials can be made with diffusion bonding. Now the contact is intimate and reliable, and the joint retains high mechanical strength at

TABLE 13-1

Powder designation	Chemical composition, %										Bulk density of powder, $\times 10^3$ kg m^{-3}	Powder hardness, Brinell, $\times 10^2$ MPa
	Cr	Ni	Cu	Si	Mn	C	S	P	Fe	Other elements		
ПР0Х18Н10			—			0.08				—	2.6-3.4	—
ПРХ18Н9	16.0-20.0	8.0-11.0		0.8	1.0	0.12			Balance	—	2.6-3.4	—
ПРХ18Н9Д7			6.0-8.0			0.10				—	2.6-3.4	—
ПРХ30С2Ю	28.0-30.0	—		2.0-2.5	—	0.08				0.8 Al max	2.6-3.4	—
ПРХ18Н10С5			—	4.5-5.5	1.0	0.12	0.02	0.035		2.5-3.5 Mo	3.0-4.0	—
	16.0-20.0	8.0-11.0							—			
ПРХ18Н10М3Д3С3				2.5-3.5		0.12				0.7 Ti max	3.2-4.0	
ПР0Х23Н28М3Д3Т	22.0-25.0	26.0-29.0	2.5-3.5	—	—	0.06-0.10			Balance		3.0-4.0	—
ПРН	—	95.0-98.0	0.6	—	—	0.10	0.03	—	—	—	3.8-4.1	—
ПНКZТ1	—	99.7	0.003	0.002	0.001	0.30	0.001	0.01	0.01	—	3.0-3.5	—
ПНЭ1	—	99.5	0.08	0.03	—	0.02	0.008	0.05	0.20	—	3.2-3.4	—
ПЖ3С	—	—	—	0.25	0.7	0.15	0.05	0.6 max	98.0-99.5	7-11 Sn	2.2-3.8	5-10
ПРБ	—	—	Balance					0.4 max	—	7-9 Sn	3.0-4.0	5-10
ПРБС	—	—							—	13-19 Pb	3.0-3.7	5-10
ПТС	—	0.4 max	—	0.07-0.10	—	0.03	—	—	—	Balance Ti	0.8-0.95	5-10
ПТМ	—	0.4	—		—	0.07	—	—	—	Same	1.7-1.8	5-10
ПТЭМ			—		—		—	—	—	Same	1.1-1.8	12-15
ПТЭК	—	—	—	0.02	—	0.02	—	—	—	Same	1.1-1.8	12-15.5
ПТЭС												

TABLE 11-2

Pair to be joined	D	δ	l	D_1	l_1
	22	3	50	—	—
	40	3	80	—	—
	40	5	100	—	—
	40	3	100	—	—
	47	5	15	—	—
	47	4	25	—	—
	60	4	30	—	—
	60	4	100	—	—
	90	5	100	—	—
	52	2	5	12	—
	52	2	7	25	—
	96	2	9	37	—
	96	3	9	37	—
	22	3	50	25	65
	40	3	100	50	800
	40	2	500	45	520
	60	4	100	50	600
	30	3	25	33	35
	16	2	20	33	35
	16	3	30	33	35
	20	3	30	40	45
	25	3	25	30	35
	16	3	30	33	35

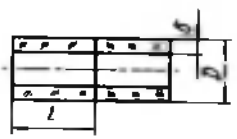



TABLE 13-1

Bonded pair	Grain size, mm	Specimen porosity, %	Air throughput, $m^3\ min^{-1}$, at Δp = 6 kPa		Pore size (avg), μm		Bursting (upsetting) pressure, MPa	
			before bonding	after bonding	before bonding	after bonding	before bonding	after bonding
ПРХ18Н9 + ПРХ18Н9	0.3	39.7	0.68	1.31	93	93	4.9	4.9
					90			
		39.5	0.70					
	0.3	40.5	0.67	1.28	106	106	4.9	4.9
	0.3	40.0	0.64		98			
		38.8	0.68	1.31	87	93	4.9	4.9
		39.0	0.69		93			
	0.4	38.6	1.30	2.26	172	165	3.4	3.9
ПРХ18Н10С5	0.6	38.0	1.20		160			
+ ПРХ18Н10С5		40.0	0.43	0.76	142	138	4.9	4.9
ПРХ30С2Ю	0.3	40.2	0.39		129			
+ ПРХ30С2Ю		40.0	0.43	0.72	149	145	4.9	4.9
		39.0	0.38		139			
	0.063	38.8	0.16	0.28	41	40	4.9	4.9
					38			
		38.0	0.15				4.9	4.9
	0.1	36.0	0.18	0.35	49	52		
ПР23Н28М3Д3Т	0.3	38.0	0.25		56			
+ ПР23Н28М3Д3Т		35.0	0.24	0.42	90	92	4.9	4.9
		35.0	0.28		96			
	0.3	38.0	0.26	0.47	90	92	4.9	4.9
ПТС + ПТС		37.0	0.30		95			
	0.063	40.2	0.67	1.24	6.3	6.3	2.9	2.9
		41.8	0.65		6.6			

Notes. 1. The test specimens were bushings 40 mm O.D., 3 mm wall thickness, and 100 mm long.

2. The air throughput for the ПРХ18Н9 + ПРХ18Н9 pair was measured at a pressure drop of 2.9 kPa.

3. The tensile tests were made at a pressure of 2.9-4.9 MPa; the specimens remained intact upon removal of the pressure.

4. Each assembly consisted of two pieces of the same dimensions, prepared from a powder of spherical particles.

The strength of joints in porous materials is most of all affected by the quality of surface preparation, especially parallelism and roughness. The out-of-parallelism should not exceed 0.05 mm. Because of this, porous materials are often joined with an interlayer of nickel (or some other fusible metal), as it facilitates the mating of the surfaces and speeds up diffusion owing to the high diffusion activity of nickel. In the course of bonding, the interlayer melts and retains its volume for the duration of diffusion treatment at constant temperature. Ordinarily, use is made of nickel foil 0.10-0.50 mm thick or a finely divided powder. Alternatively, an interlayer can be electroplated or evaporated in vacuum (in which case its thickness is 0.01-0.05 mm). The mating surfaces are machined with the workpieces clamped in a suitable mandrel or arbour. Prior to machining, the pieces should be put together in a suitable jig for proper alignment and in order to keep the clearances to a minimum (see Fig. 13-1).

Porous materials can be bonded on any type of unit capable of maintaining the requisite temperature and of applying the correct bonding pressure. Heating must be localized and intensive, but without causing surface fusion. The bonding opera-

TABLE 13-4

Sintered pair	Grain size, mm	Porosity, %	Bonding temperature, K	Bonding pressure, MPa	Holding time, min	Minimum bursting (internal) pressure, MPa
ПР0Х18Н10 + ПР0Х18Н10	0.3	40	1423-1473	2-3	1.0-1.5	4.9
ПРХ18Н9 + ПРХ18Н9	0.3	40	Same	2-3	1.0-1.5	4.9
ПРХ18Н9Д7 + ПРХ18Н9Д7	0.4	38	Same	2-3	1.0-2.0	4.9
ПР0Х23Н28М3Д3Т + ПР0Х23Н28М3Д3Т	0.3	38	1523-1573	2-3	1.0-1.5	2.0
ПРХ30С2Ю + ПРХ30С2Ю	0.6	38	1523-1553	2-3	1.5-2.5	2.0
ПРХ18Н10С5 + ПРХ18Н10С5	0.4	40	Same	2-3	1.5-2.5	2.0
ПНК2Т1 + ПНК2Т1	0.3	40	1373-1423	10-20	0.5-1.0	1.5
ПЖЗС + ПЖЗС	0.4	30	1473-1523	30-50	3.0-5.0	4.9
ПТЭМ + ПТЭМ	0.063	40-45	1203-1233	15-25	1.0-1.5	2.0
ПТЭК + ПТЭК	0.4	38-40	1223-1273	10-20	1.0-1.5	2.0

Note. The specimens remained intact upon removal of the test pressure.

tion should be carried out in a vacuum of at least 2.66×10^{-1} MPa in a neutral atmosphere (argon or hydrogen).

In the diffusion bonding of porous materials, it is essential to secure the same porosity (in terms of the size and number of pores and their permeability by fluids) both in the bond and in the parent material. This can only be achieved if the degree of contact between particles is the same throughout the material. In addition to proper contact, this is also essential to assure the same strength and filtering capacity of the product.

As has been found [1], the maximum contact fraction in a porous sintered material is $(1 - n)^3$, where n is the porosity in fractions of a unit. Therefore, in calculating the bonding pressure, the porosity of sintered products must be taken into consideration. As the porosity increases, the bonding pressure should be reduced. For coarse-grained powders the bonding temperature must be raised by 293-323 K. The duration of pressure will sometimes depend on the quality of the sintered material. A more loosely sintered material will usually require a more rigorous heating. The characteristics of some bonded products are given in Table 13-3, and the suggested bonding variables for some materials are listed in Table 13-4.

13.3 Bonding of Porous Materials to Steels

Joining by bonding is a frequent occurrence in the fabrication of complicated shapes. Diffusion bonding is especially advantageous when a porous material is to be joined to a steel shank or backer. As in all other cases of diffusion bonding, joint strength depends on the quality of surface preparation (notably, roughness) and the

TABLE 13-5

Sintered pair	Bonding temperature, K	Bonding pressure, MPa	Holding time, s	Minimum breaking load, MPa[1]
ПРХ18Н9 + 12Х18Н9Т	1473-1523	2.0×10^4-3.0×10^4	120-180	5.065×10^6
ПРХ18Н9 + 2Х13	1473-1523	Same	120-180	Same
ПРХ30С2Ю + 12Х18Н9	1523-1553	Same	120-240	Same
ПРН + 12Х18Н9	1423-1473	Same	60-120	3.039×10^6
ПНЭ1 + 12Х18Н9	1423-1473	Same	60-120	Same
ПЖ3С + steel-45	1523-1573	3.0×10^4-5.06×10^4	180-300	Same
ПТС + ВТ1	1173-1223	1.51×10^4-2.53×10^4	120-180	2.026×10^6
ПТМ + ОТЧ	1173-1223	Same	120-180	Same
ПТЭК + ВТ1	1173-1273	Same	120-180	Same

[1] The fracture occurred in the porous materials; no failure took place at the bond.

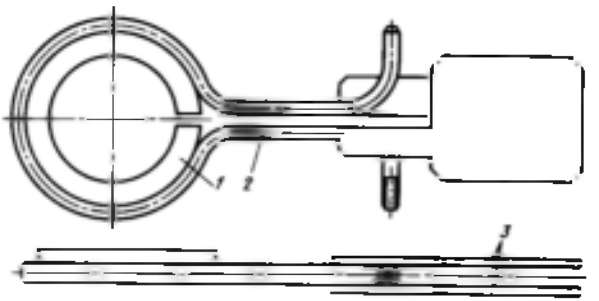


FIG. 13-2 Inductor for the diffusion bonding of a porous material
1 — copper ring; *2* — copper tube 6 mm dia.; *3* — copper plate

parallelism of the mating faces. The faces of the pieces to be joined must be parallel to within 0.2 mm. Another factor affecting joint strength is the choice of the bonding variables. The recommended values for joining porous materials to steels are listed in Table 13-5.

There is a marked difference between porous materials and steels in thermal expansion, thermal conductivity and ultimate strength. The difference in thermal expansion inevitably leads to internal stresses which distort the components and may be responsible for cracking or even complete failure. This is why it is essential to minimize the magnitude and character of internal stresses. One way to do this in joining a porous material to a steel is to use local heating.

Experience shows that the necessary heat can be best supplied by pulses of h.f. current [4, 7]. The steel piece of the assembly should first be raised to glow (873-973 K), following which the inductor should be moved 1 or 2 mm to one side so as to heat the porous part. The shape of the inductor used for joining porous cermets to steels is shown in Fig. 13.2. As is seen, the inductor is a single-turn flat ring 3 mm thick, with a tube brazed to one side in order to cool the inductor with running water. The inductor is held stationary on the outer side of the parts being joined.

13.4 Performance of Diffusion Bonds in Porous Products

Porous tubes joined by diffusion bonding have shown good performance in filters for fluids. In an experimental filter the tubes were made of heat-resistant steel powder with spherical particles and with pores measuring 100-120 μm and 200-300 μm. Seven tubes of one size were used in each set. The tubes were 500 mm long and 40 mm in diameter so that the filtration area was anywhere between 0.35 and 0.45 m^2.

Each set was allowed to operate until the filtration rate had dropped to a predetermined limit, following which the filter elements were regenerated by blowing back with compressed air under a pressure of 4.052×10^5 MPa for about 30 s.

Such cycles were repeated until the tubes had served their specified service life. At the end of the tests, none of the diffusion bonds had failed, and the filter elements were sufficiently strong for further use.

When subjected to bending tests, the tubes resisted a load of 200-300 kg, and only failed in the parent material when loaded with 400-500 kg; the diffusion bonds remained intact. The porosity was 40% as it had been before. The filter elements consistently caught mechanical impurities with a particle size of 0.2 μm; the filtration efficiency was 99.6%.

Diffusion-bonded porous products serve equally well in gas-analysis apparatus. Also as already noted at the beginning of this chapter, a porous element can conveniently be joined to a solid steel backer to make a reliable flame barrier, so that the instrument design can be simplified and made less expensive. The joint between the porous member and the steel flange is air-tight and mechanically strong. The assembly can stand up to a test pressure of over 2.026×10^6 MPa.

Diffusion bonding has been used to make small-size silencers for hand-operated pneumatic tools with spent air exhaust into the handle. In a trial, the joints between the porous elements and metal backers were tested for strength and tightness. In a stress-rupture test, the products remained intact at a load of over 2-3 MPa [2], and the bond showed good tightness as the pore size in the assembly was 130 μm.

From the foregoing it is seen that the use of diffusion bonding in powder metallurgy is extremely beneficial. Apart from the cases quoted above, it can be used to fabricate far more elaborate and sophisticated products of high practical value.

Bibliography

1. BALSHIN, M. Yu., KIPARISOV, S. S. *The Foundations of Powder Metallurgy.* Metallurgiya, Moscow, 1978, pp. 18-58; 123-145 (in Russian).
2. *Diffusion Bonding of Porous Noise Silencers.* Abstracts of Reports at the 9th All-Union Scientific-Engineering Conference on the Diffusion Bonding of Metals and Nonmetals. Moscow, 1979, p. 60 (in Russian).
3. KAZAKOV, N. F., *Diffusion Bonding of Materials.* Mashinostroyeniye, Moscow, 1976 (in Russian).
4. KAZAKOV, N. F., SOLUYANOVA, Ye. K., SHIBRYAEV, B. F. *The Diffusion Bonding of Highly Porous Materials.* "Svarochnoye proizvodstvo", 1976, No. 7, pp. 16-18 (in Russian).
5. *Porous Sintered Sound-Absorbing Elements.* TsINTIkhimneftemash, Moscow, 1978 (in Russian).
6. *The Use of Induction Heating in Powder Metallurgy.* Proceedings of the 7th All-Union Scientific-Engineering Conference on Powder Metallurgy. ERPI, Yerevan, 1964 (in Russian).
7. SHIBRYAEV, B. F., PAVLOVSKAYA, Ye.I. *Cermet Filter Elements.* Mashinostroyeniye, Moscow, 1972, pp. 95-100 (in Russian).

14

Diffusion Metallurgy

A.V. Sergeyev

Diffusion bonding has been gaining an ever wider ground in the fabrication of new composite materials. In fact, it is the most versatile process that can be used for the purpose. The closely controlled process variables (temperature, pressure, loading rate, duration of pressure, etc.) assure the production of composites with the desired and reproducible properties and qualities.

Diffusion bonding is used to make both semi-finished and finished products from composite materials. The fabrication process is similar to the diffusion bonding of two members and includes surface preparation, heating in vacuum, a shielding or a reducing atmosphere, application of bonding pressure (pressing load), diffusion treatment at a constant temperature, and cooling.

A composite material (or, simply, a composite) is a laminate made up of two or more plates of dissimilar materials bonded together. Through an appropriate selection and design, it is possible to make a composite having the desired corrosion resistance, surface hardness, wear resistance, strength, thermal conductivity, electric and magnetic properties, controlled deformation under varying temperature (so-called thermostat bimetals), etc. The use of a multilayer design serves to enhance resistance to brittle failure. A sizeable economy of nickel-base steels can be achieved by using ferritic steels for cladding. For example, cladding steel-45 (a carbon steel) with 0.9 mm of 12X18H10T steel improves the low-temperature impact toughness of the base material and brings down its critical brittleness.

The usual method of producing bimetal or multilayer metal composites is, firstly, to make a "sandwich" of an appropriate thickness from two or more plates and, secondly, to roll the sandwich to the desired final thickness. Taking the Soviet-made thermostat bimetals ТБ 2013 and ТБ 1613 as an example, the bonding variables are as follows: temperature, 1 123 K; bonding pressure, 19.6 MPa; duration of treatment at constant temperature (holding time), 10 min; and vacuum, 6.6×10^{-2} Pa. Sometimes, the plates to be made into a composite are electroplated, evaporated, etc. with another material.

Fibre composites are fabricated by diffusion bonding, using matrices of aluminium, titanium, nickel, iron, cobalt, magnesium and some other metals and alloys. Most frequently, the matrix is in the form of foil. The necessary reinforcement is supplied by whiskers (short single crystals), fibres (multicrystalline structures), or wire. Prior to diffusion bonding, the reinforcing elements (whiskers, fibres, or wire) are usually degreased by a chemical means; the foil is washed, degreased, cleaned mechanically, and treated chemically. The reinforcement is placed in alternating layers with the matrix material in the form of fabric, gauze, or individual fibres. The fabric and gauze are made from metal fibres or graphite. Highly

brittle single fibres are wound before they are inserted in the matrix. The multilayer tape thus made is then plasma-sprayed and the sandwich is made by diffusion bonding. Alternatively, fibres can be secured in place by cementing, but this entails the contamination of the material.

In making a composite in which the matrix is nickel and the reinforcement is carbon fibres, it is usual to electrodeposit nickel onto the fibres. The recommended formulation for the electrolyte (in grams per litre) is as follows: nickel sulphite, 150; ammonium chloride, 15; and boric acid, 15. A uniform nickel plating is usually obtained with a current density of 1 A dm^{-2}. Nickel-plated carbon plates are then diffusion-bonded at a temperature of 1273-1573 K and a bonding pressure of 19.6-29.4 MPa. According to a Japanese source, a higher bonding temperature is required as the bulk fraction of the reinforcing component is increased.

The starting material for diffusion bonding can be boron-fibre-reinforced, boron-nitride-plated tape impregnated with molten aluminium. For the composite to have a sufficient strength (which obeys the law of additivity), a reliable mechanical bond must exist at the interface. When this condition is met, the matrix will reliably transfer load to the fibres in service. On the other hand, the components of a composite interact with one another. Diffusion processes diminish the strength of the reinforcing phase and lead in most cases to the formation of an intermetallic layer at the interface between the fibres and the matrix. When the intermetallic layer grows 0.5-2.0 μm wide, the composite ceases to exist. Now, when loaded, the matrix will not transfer the stress to the fibres, the intermetallics will be dispersed, and cracks will form and propagate in the fibres. The formation of solid solutions does not impair the quality of a composite in a drastic way.

The high-temperature strength and service life of a composite can be enhanced by applying a diffusion barrier to the fibres. Such a barrier can prevent or markedly slow down the interaction between the fibres and the matrix. A diffusion barrier should be applied so that it forms a strong bond with the fibres, has the same thickness throughout, and is free from porosity. An alternative method for suppressing the formation of undesirable phases at the interface is to make the matrix from alloys having a low reactivity towards the reinforcement material. From a thermodynamic point of view, it is essential to keep the difference in chemical potential between the components of a composite to a minimum.

In a composite with an aluminium matrix reactions are retarded by the naturally occurring oxide film on the aluminium. The oxide film will retain its diffusion barrier properties, if diffusion bonding does not cause cracking in the film, and this requires that diffusion bonding be conducted at optimal temperature and bonding pressure.

A diffusion bonding unit adapted to fabricate composites usually includes a press, a vacuum system, and a heating facility. Composites are diffusion-bonded primarily in moulds and between heated platens. The starting materials in the form of sheets or long tape are prepared between heated platens stepwise. At first, a diffusion bond is made within the area nearest to one end of the sandwich, then the sandwich is advanced, and another diffusion bond is made within the adjacent area along with some of the previously bonded area.

TABLE 14-1

Matrix	Fibre	Fibre fraction (% by volume)	Bonding temperature, K	Bonding pressure, p_b, $\times 10^{-1}$ MPa	Holding time, min	Ultimate tensile strength, σ_{tr}, $\times 10^{-1}$ MPa
Aluminium	Boron	50	853	294	2	107
Aluminium alloys:						
7178	Boron	40	727	411	60	137.2
6061	Boron	48	763	343	60	117
2024	Boron	30	773	475	60	56-81
Magnesium	Boron	25	798	686	60	86-95
		26	773	1372	60	46

Alternatively, bonding pressure in the fabrication of composites can be applied by isostatic (autoclave) pressing or dynamic pressing. In the case of isostatic pressing, the bonding pressure is applied by a gas contained in a high-pressure cylinder. Dynamic pressing is a pulse method. Now the bonding pressure is applied to the preheated sandwich for a very short span of time.

The process variables for the fabrication of composites and their ultimate tensile strength are listed in Table 14-1 [2].

Sometimes composites are made from powdered materials by a combination of sintering and diffusion bonding. At first, a blank is shaped from raw powders. Then the shape is both sintered and diffusion-bonded to a backer. To avoid discontinuities, the bonding pressure is kept at a value which will not cause a marked distortion or departure from the desired dimensions, or the bonding is done in suitable compression moulds. This combination of sintering and diffusion bonding is applicable to components made up of a solid metal portion and a powdered (metallic or nonmetallic) portion.

An important aspect in diffusion metallurgy is heat treatment in a vacuum. This may be a separate step or one combined with the diffusion bonding proper. As compared with heat treatment in controlled atmospheres, vacuum heat treatment offers several advantages: the atmosphere is extremely pure and homogeneous; the physical and engineering properties of the materials are improved by a wide margin; undesirable impurities such as components with a high vapour pressure (magnesium and manganese) are sublimed and removed; temperature- and pressure-dependent chemical reactions are speeded up; carburization and decarburization are avoided. Many alloys based on such metals as titanium and niobium are heat-treated in vacuum only.

Vacuum heat treatment can be used to clean the mating surfaces prior to bonding, to harden, temper or anneal the material, and to improve the physico-chemical and mechanical properties of the materials and joints.

Vacuum heat treatment for cleaning involves removal of dissolved gases, reduction (dissociation) of oxides on the surfaces, and pyrolysis of contaminants.

The gases dissolved in metals may be classed into three groups: (1) those physically adsorbed (mainly air components and hydrocarbons); (2) those on the sur-

face in the form of oxides and other chemical compounds; and (3) gases in the bulk. When thermally degassed, most metals give up primarily hydrogen. In the case of steel, tungsten, and molybdenum, it is also necessary to consider nitrogen. Oxygen is thermally degassed in minute quantities.

The quantity of gases adsorbed on a surface can be reduced by giving the surface a better finish and by protecting it against oxidation. When a smooth surface is heated in vacuum to 473-573 K, physically adsorbed gases are desorbed in a matter of a few minutes. Removal of adsorbed gases from rough or oxide-coated surfaces calls for a higher temperature. Importantly, the rate of heating must be coordinated with the rate of gas withdrawal. Failure to do so might lead to chemisorption and chemical reactions (the formation of oxides, nitrides, and hydrides).

The temperature and time required for removal of gases from the bulk material by vacuum heat treatment are chosen subject to several considerations. The temperature ought not to cause vaporization or, less so, fusion of the metal. The maximum temperature should be such that the components experience no distortion (on approaching the yield limit). For unstressed components, it must be close to the annealing temperature; for stressed parts, below that point. For most metals, the recommended temperature lies in the range 1173-1273 K. Molybdenum, tungsten and tantalum require a higher temperature (half the gas absorbed in molybdenum can be removed only by heating to above 1 473 K). The temperature for titanium and copper ranges between 773 K and 973 K. The quantity of gas present in components will be markedly reduced, if they are baked out in two steps: first at a low temperature (673-773 K), and then at a higher temperature (1123-1273 K) for bulk degassing.

When exposed to a high vacuum, metals undergo a change in mechanical properties, notably an increase in ductility.

When single and polycrystals of aluminium are subjected to tension in a vacuum of 1.33 MPa, the elongation to failure increases by 40%, but the ultimate tensile strength decreases. It has been observed that vacuum also affects the mechanical properties of magnesium and molybdenum. The effect can be attributed to the formation of an oxide film which serves as a barrier for the emergence of dislocations. The dislocations concentrate in the subsurface layer, and the material gains in hardness. If the vacuum is sufficiently high to prevent the formation of an oxide film on the clean surfaces, the emergence of dislocations is facilitated, and the ductility of

TABLE 14.2

Material	Heat treatment	Pressure, Pa	Temperature, K
High-speed steel	Hardening	1×10^{-1}	1533
Same	Tempering	1×10^{-2}-1×10^{-3}	813
Corrosion-resistant steel	Hardening	1×10^{-1}-1×10^{-2}	1323-1373
Tantalum	Annealing	1×10^{-3}	1703
Titanium	Annealing	1×10^{-2}-1×10^{-1}	503-1020
Zirconium	Annealing	1×10^{-2}-1×10^{-1}	873-973

the material is improved. There is a further improvement in ductility owing to removal of gas impurities from the surface layer upon deformation.

Typical conditions for heat treatment in vacuum are listed in Table 14-2.

Heat treatment in vacuum reduces porosity in welded joints. It can heal the damage caused in a metal at the early stages of fatigue failure.

Bibliography

1. KREIDER, K. (ed.). *Composite Materials*. Mashinostroyeniye, Moscow, 1978, vol. 4 (in Russian).
2. PORTNOI, K. I. , SALIBEKOV, S. Ye. , SVETLOV, I. L. *The Structure and Properties of Composite Materials*. Mashinostroyeniye, Moscow, 1979 (in Russian).
3. KAZAKOV, N. F. *Diffusion Bonding of Materials*. Mashinostroyeniye, Moscow, 1976 (in Russian).
4. KAZAKOV, N. F. , LADO, L. N. *The Formation of a Diffusion Bond Between Powdered Materials and a Low-Carbon Steel*. "Avtomaticheskaya svarka", 1971, No. 11, pp. 65-66 (in Russian).
5. KAZAKOV, N. F. , MUSIN, R. A. *A Study into the Mechanism of Diffusion Bonding of Zinc Sulphide to Metals*. In: "Diffusion Bonding in Vacuum", PNILDSV, Moscow, pp. 206-214 (in Russian).
6. KAZAKOV, N. F. *Improvement of Metals and Alloys by Vacuum Heat Treatment*. In: "Diffusion Bonding in Vacuum", PNILDSV, Moscow, 1971, pp. 204-206 (in Russian).
7. FRANTSEVICH, I. N. , KARPINOS, D. M. *Production of Composites by Vacuum Diffusion Bonding and Rolling*. In: "Diffusion Bonding in Vacuum", PNILDSV, Moscow, 1970, pp. 299-307 (in Russian).

15

Nonmetals and Their Joining to Metals

V. P. Antonov and *V. A. Bachin*

15.1 General

Advances in present-day science and technology are inseparably related to the knowledge and use of materials widely differing in properties — high-temperature, heat-resistant, superconducting, magnetic, corrosion-resistant, current-conducting, insulating, chemically and radiation resistant and so on.

All crystalline inorganic substances may be classed into metals and nonmetals. Furthermore, they may be grouped into conductors, semiconductors, and dielectrics (Table 15-1).

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Bibliography

1. KREIDER, K. (ed.). *Composite Materials*. Mashinostroyeniye, Moscow, 1978, vol. 4 (in Russian).
2. PORTNOI, K. I. , SALIBEKOV, S. Ye. , SVETLOV, I. L. *The Structure and Properties of Composite Materials*. Mashinostroyeniye, Moscow, 1979 (in Russian).
3. KAZAKOV, N. F. *Diffusion Bonding of Materials*. Mashinostroyeniye, Moscow, 1976 (in Russian).
4. KAZAKOV, N. F. , LADO, L. N. *The Formation of a Diffusion Bond Between Powdered Materials and a Low-Carbon Steel*. "Avtomaticheskaya svarka", 1971, No. 11, pp. 65-66 (in Russian).
5. KAZAKOV, N. F. , MUSIN, R. A. *A Study into the Mechanism of Diffusion Bonding of Zinc Sulphide to Metals*. In: "Diffusion Bonding in Vacuum", PNILDSV, Moscow, pp. 206-214 (in Russian).
6. KAZAKOV, N. F. *Improvement of Metals and Alloys by Vacuum Heat Treatment*. In: "Diffusion Bonding in Vacuum", PNILDSV, Moscow, 1971, pp. 204-206 (in Russian).
7. FRANTSEVICH, I. N. , KARPINOS, D. M. *Production of Composites by Vacuum Diffusion Bonding and Rolling*. In: "Diffusion Bonding in Vacuum", PNILDSV, Moscow, 1970, pp. 299-307 (in Russian).

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All crystalline inorganic substances may be classed into metals and nonmetals. Furthermore, they may be grouped into conductors, semiconductors, and dielectrics (Table 15-1).

TABLE 15-1

Class	Group	Materials	Application
Metals	Conductors	Metals Alloys Steels Cemented carbides	Tools, blades, rotors, stators, magnets, etc.
	Semiconductors	Simple semiconductors	Oscillators, amplifiers, rectifiers, converters, magnetic devices, ferroelectric devices, catalysts
		Complex semiconductors, their solid solutions Ferrites Alpha-quartz Ferroelectrics Metal oxides	
Nonmetals	Dielectrics	Glass	Sight glasses, shields, vacuum lead-ins, envelopes
		Pyrosils	Insulator assemblies, r.f. devices
		Ceramics	Vacuum and gas-filled tubes, atomic reactors, particle accelerators, chemical reactors
	—	Graphite	Packing seals, current leads

In engineering, the need to join nonmetals to metals and alloys is a frequent occurrence. The joints are required to be mechanically and electrically strong under static and dynamic loads, vacuum-tight, thermally durable, and stable towards radiation, to resist attack by various corrosive media, to retain their original dimensions and shape, etc. These objectives can be achieved by joining in any one of several ways as listed in Table 15-2.

No matter how sophisticated the techniques and joint design may be, brazing will not always produce a quality joint between a semiconductor, glass or ceramic material, on the one hand, and metals and alloys, on the other. As it varies in thickness and composition, the filler material can give rise to additional internal stresses, and this will substantially impair the thermal endurance of the joint and increase electric, heat and r.f. losses. An increase in brazing temperature or holding time causes the metal plating to dissolve in the filler material, so the joint quality is further degraded. Evaporation of the filler material onto nonmetallic materials leads to an electric break-down and leakage. Practical experience and relative

TABLE 15-2

Assembly	Joining method
Metal-semiconductor	Cementing, brazing, resistance brazing, sealing-in, fusion welding (resistance, pressure, ultrasonic, electron-beam, laser-beam)
Metal-ferrite	Brazing
Metal-glass, metal-pyrosil	Sealing-in, brazing
Metal-ceramic	Brazing of metal-plated ceramic components, resistance brazing, joining with glazes and glass cements, pressure welding
Metal-graphite	Brazing, fusion welding, electron-beam welding

studies have shown that by far the best method to join semiconductors, glasses, and ceramics to metals and alloys is by diffusion bonding. Diffusion-bonded joints are used in most critical instruments and machines.

15.2 Glass-Metal Seals

Metal-to-glass joints, or seals, made by diffusion bonding are remarkably strong, stand up well to temperature variations, and can be made to close dimensional tolerances. A diffusion-bonded seal is obtained by virtue of microplastic deformation either in a soft interlayer or in one of the pieces being joined (the glass or the metal one). The degree of deformation is so negligible that the shape of the pieces is changed only slightly.

A good glass-metal seal is formed owing to the interaction between the lower oxides on the metal and the oxide system on the glass. Therefore, in designing a procedure for making a glass-metal seal the objective usually is to produce a suitable oxide layer on the metal surface and to choose conditions that will promote the above interaction with the formation of a permanent joint.

The basic component of most glasses is silica, so they are called silica glasses. A glass can be imparted some special properties by adding what are called fluxes or

TABLE 15-3

Glass	Chemical composition, parts (mass)							
	SiO_2	Na_2O	CaO	MgO	Al_2O_3	PbO	K_2O	B_2O_3
Soda-lime	72	15	9	3	1	—	—	—
Lead-alkali	68	10	1	—	—	15	6	—
Borosilicate	80	4	—	—	2	—	—	14

TABLE 15-4

Designation	Softening point, K	Coefficient of thermal expansion, α, $\times 10^{-7}$ K^{-1}, at 293 K	Porosity, %	Ultimate strength, MPa		
				compressive	bending	tensile
KB	1433	5.5	0.0	6.5	11	6
KУ	1433	5.5	0.0	6.5	11	6
KB-P	1433	5.5	0.0	6.5	11	6
KИ	1493	5.5	0.0	6.5	11	6
Extra-pure:						
type I	1573	—	0.0	6.5	11	6
type II	1373	—	0.0	6.5	11	6
Ceramic	1373-1423	5.8	5-15	3.0-4.0	0.4-0.5	2

modifiers. According to these additions, glasses may be divided into several groups each possessing specific properties (Table 15-3).

PRINCIPAL PROPERTIES OF SEALING GLASSES. *Quartz glass.* This is a single-component variety of silica glass — it consists practically of only silica. It is made by fusing pure naturally occurring varieties of silica (such as rock crystal, vein quartz or quartz sand), artificial quartz crystals, and synthetic silica. Frequently it is called fused silica glass. It possesses an extremely valuable combination of physico-chemical properties, such as refractoriness, thermal endurance, high dielectric strength, high acoustical properties, transparence to a broad spectrum of ultra-violet, visible, infra-red and radio-frequency radiation, high optical homogeneity, a minimal temperature coefficient of linear expansion, etc.

Quartz (or fused silica) glass comes in several basic varieties, such as opaque, transparent, industrial, optical, extra-pure, ceramic, and alloyed. The physical properties of several grades of Soviet-made fused silica glass are listed in Table 15-4.

At a temperature of over 1323-1473 K, quartz glass loses its elasticity and becomes plastic. Owing to the very low coefficient of thermal expansion, quartz glass has an extremely high thermal endurance.

Quartz products can be used at temperatures not over 1273-1373 K. Over 1473 K, quartz glass crystallizes into alpha-crystobalite.

Optical glasses. These are clear, transparent and homogeneous grades of glass. Each grade has a particular chemical composition, and this fact is usually reflected in the type designation it is assigned. An example of a Soviet-made optical glass is K-8 (Kron-8).

Glasses for electron devices. The manufacture of electron devices uses a wide variety of glass grades each serving a specific purpose. Some grades have been adapted to produce a reliable seal to metals. To this end, they are made with a coefficient of thermal expansion very close to that of metals and alloys. The most commonly used grades of glass for electron devices and some of their physical properties are listed in Tables 15-5 and 15-6.

TABLE 15-5

Glass grade	Coefficient of thermal expansion, α, $\times 10^{-7}$ K^{-1}, at 293 K	Density, kg m^{-3}	Young's modulus, MPa
ЛК-4	51	2330	64900
ЛК-5	35	2270	69800
ЛК-6	83	2300	49800
ЛК-7	44	2300	69300
К-5	74	2470	78900
К-8	76	2520	82300

TABLE 15-6

Glass grade	Coeff. of thermal expansion, α, $\times 10^{-7}$ K^{-1}, at 293 K	Young's modulus, MPa	UTS, MPa	UCS, MPa	Softening point, K
C47-1	47.0	63000	90.0	1090	863
C48-1	48.0	64500	77.0	1080	828
C48-2	48.0	—	—	—	843
C48-3	48.0	—	—	—	1083
C49-1	49.0	62500	30-50	—	853
C49-2	49.0	66700	30-50	850	858
C49-3	49.0	—	—	—	788
C50-1	50.0	—	—	—	893
C50-2	50.0	—	—	—	838

Preparation of glass for bonding. Machining leaves a network of fine cracks on the surface of a glass component. These cracks might substantially impair the strength of both the glass and of the bond (seal) made to it. This is why the fissured layer should be removed. This can be done by treating the glass with a solution which consists of 4 (wt) parts of concentrated hydrofluoric acid, 60 (wt) parts of water, 33 (wt) parts of concentrated nitric acid, and 2 (wt) parts of a surfactant. The solution should be used at a temperature of 293 K. The relative amounts of the acids may be varied as appropriate. A solution with too much of hydrofluoric acid tends to etch away the glass. After the treatment with the solution the glass must be thoroughly rinsed in running water.

If the etching of the glass surface is objectionable, proceed as follows. Boil the glass component in a 5-% solution of hydrogen peroxide, add some NH_4OH so that the solution has pH 11.0; rinse the component thoroughly in tap, then in distilled water; and allow the part to dry, preferably in a clean atmosphere. The above solution does not produce an etching effect on glass.

TABLE 15-7

Metal or alloy	Melting point, K	Coefficient of thermal expansion, α, $\times 10^{-7}$ K^{-1}, at 293 K	Young's modulus, E, $\times 10^{-3}$ MPa	Ultimate tensile strength, MPa
Tungsten	3683	44.4	365-400	1800-4150
Molybdenum	2933	55	280-330	700-1000 1400-1800
Tantalum	3269	65	186-190	350-1200
Zirconium	2225	60	90-100	200-1000
Titanium	1943	82	98-117	450-1020
Platinum	2044	90.7	100-175	140-370
Palladium	1825	119	112-130	140-540
Iron	1804	125	207-217	180-620
Nickel	1725	133	180-227	320-1000
Copper	1356	165	117-130	160-500
Aluminium	933	238	66-73	80-110
Platinite	—	60-65 80-100	—	280-380
08X18H10T steel	1698	173	190-200	560-630
Kovar	1723	50	—	540

Of the two methods of surface preparation, the first is more efficient, as it removes fissures along with a thin surface layer of the material, thereby improving its strength. It should be remembered that this method ought not to be applied to optical elements as it impairs their transparency.

SEALING METALS AND ALLOYS. For a proper metal-to-glass seal, it is important to match the two materials in thermal expansion. Otherwise, the stresses produced by temperature variations will cause the seal to fail. In the Soviet Union, the most commonly used sealing metals are Fe-Ni alloys, Kovar and stainless steel, and also the pure metals copper, nickel, titanium, aluminium, molybdenum, tungsten, and some others. The physical properties of some sealing metals and alloys are listed in Table 15-7.

The stresses produced in sealing alloys by preparatory machining can readily be relieved by annealing in vacuum. Vacuum is essential because sealing alloys are susceptible to intergranular corrosion. Large and bulky components should be annealed at 1323-1373 K for 10 min; small and thin-walled components should be annealed at 1 223 K for 10 min. On Fe-Ni alloys, an oxide coating can be produced by allowing them to cool after they have been annealed in vacuum and air has been submitted to the vacuum chamber. The oxide coat forming on the surface of Fe-Ni alloys is a mixture of the oxides of nickel, chromium, and iron. When properly oxidized, the surface will be green in colour. In the case of overoxidation, the surface may be dark-brown or even black.

Kovar. This is a sealing alloy admirably matching a wide range of glasses in thermal expansion. It can successfully be sealed to optical glasses with a coefficient of thermal expansion in the range 45×10^{-7}-55×10^{-7} K^{-1}. A distinction of Kovar is that it has a very high Curie point which almost coincides with the transformation point of borosilicate glasses.

Prior to diffusion bonding, a Kovar component should be treated as follows:

— degrease it in carbon tetrachloride or some other organic solvent;

— immerse it for 1-3 min in concentrated hydrochloric acid raised to 333-353 K;

— rinse in an alkaline solution, then in cold tap water, and finally in distilled water;

— dry it with ethanol or methanol, and allow it to dry in an oven.

The above treatment should be applied immediately before diffusion bonding (sealing). Thus treated, the parts should never be handled with bare hands. The surface of Kovar pieces should be oxidized prior to diffusion bonding in order to make a strong gas-tight seal. For better adhesion of oxides, overpolishing of the parts should be avoided. The surface of the parts should be gray and display no metallic lustre. After sealing, the bond may be from light-gray to brownish-gray in colour, depending on the grade of glass and the thickness of the oxide coating. The appearance of metallic lustre is an indication of underoxidation, whereas an excessive blackening is a sign of overoxidation of the metal surface.

Copper. A wide gamut of valuable properties make copper a good sealing material for glass-metal seals in which it may serve either as a structural member or as an interlayer. Copper interlayers go a long way towards making good seals of quartz to quartz and to other materials. Because copper has a very high coefficient of thermal expansion, there are no sealing glasses that would match it perfectly. Because of this glass-copper seals are made by techniques permitting a degree of mismatch. Most reliable seals are obtained when the surface of the copper is coated with Cu_2O. To form this coating, the assembly is loaded in a vacuum chamber, the space is evacuated to a pressure of 0.133 Pa, and the work is raised to a temperature of 1 223 K. On holding the assembly for 10 minutes it is allowed to cool to 573 K at which temperature air is admitted to the chamber. This treatment produces a uniform coat of red copper oxide.

Aluminium. Being highly plastic and highly reactive, aluminium is widely used in diffusion-bonded seals for complex glasses and pure quartz. It can be used both as a structural member of a seal and as an interlayer. A good proportion of the stresses arising in glass-aluminium seals can be removed by relaxation occurring when the seal is allowed to cool slowly after making. As experience shows, however, a cooling rate of 10 K min^{-1} may sometimes prove ineffective. The aluminium part of a seal should be prepared for bonding as follows:

— degrease it with an organic solvent (if necessary);

— immerse it in a 10-% solution of NaOH at a temperature of 333 K for 3-5 min;

— immerse it finally in a brightening solution consisting of 2 weight parts of H_2SO_4, 1 weight part of HNO_3, and 1 weight part of H_2O.

PROCEDURES FOR MAKING GLASS-METAL SEALS BY DIFFUSION BONDING. In designing a procedure, the process variables involved can best be chosen by trial and error.

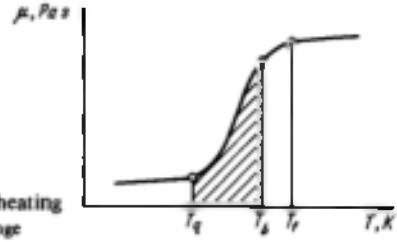

FIG. 15-1 Variations in the viscosity of glass with heating
T_g-T_f, vitrification range; T_g-T_b, bonding range

A typical curve illustrating variations in the viscosity of glasses on heating is shown in Fig. 15-1 where the shaded area defines the temperature range for diffusion bonding.

For ЛК-4 glass it has been found experimentally that at a temperature of 823 K the glass begins to deform under a bonding pressure of over 5 MPa, whereas at a temperature of 773 K the critical bonding pressure rises to 14.5 MPa. For glasses of Kron-8 type the region of initial deformation under load is shifted towards higher temperature and lies anywhere between 853 K and 893 K. In order to determine the bonding temperature for a specific grade of glass it is therefore necessary to determine the temperature at which the applied bonding pressure brings about the onset of plastic deformation. The bonding pressure must be high enough to produce the requisite microplastic deformation at the seal, at least sufficient to establish a complete contact between the mating surfaces. As experience has shown, with a correctly chosen temperature the bonding pressure is 2-8 MPa. In the circumstances, diffusion treatment at constant temperature (the holding time) for 20-40 mm proves sufficient for microplastic deformation to establish contact over the entire area of the seal.

Frequently, reliable glass-to-glass and glass-to-metal seals are made with the use of metal interlayers which may serve as activators for the mating surfaces (owing to the higher ductility of the interlayer in comparison with the base material); as compensators for the stresses arising when a seal involves materials differing in thermal expansion; as accelerators for mass transfer or chemical reactions; and/or as buffers to prevent the formation of undesirable phases.

Most often, interlayer materials are aluminium, copper, Kovar, niobium, and titanium, in the form of foil usually not over 0.2 mm thick. Thicker interlayers can act as structural members in their own right. In the vacuum diffusion bonding of seals involving quartz, the effect of residual stresses on the strength characteristics of the assembly can be minimized by using interlayers not more than 0.05 mm thick.

The choice of the vacuum to be used depends on the properties of the interlayer used. It is important to remember that the making of glass-to-metal seals differs from the diffusion bonding of metals — now the presence of an intermediate oxide coating is essential for proper bonding. A very deep vacuum can change the stoichiometry of the original oxide coating on the metal. When quartz seals are made with a red copper oxide coat present at a vacuum deeper than 0.0133 Pa, the interlayer eagerly dissociates, and the strength of the seal is impaired. In air, no seal

TABLE 15-8

Materials joined	Interlayer	Bonding conditions[1]		
		temperature, K	pressure, MPa	holding time, min
Quartz + quartz	Al	893	12	40
Quartz + quartz	Cu	1223	10	30
Quartz + quartz	Ni	1423	12	40
Quartz + quartz	None	1423	8	30
C49-2 + Kovar	None	863	5	20
ЛК-4 + Mo	Al-Ni	803	10	40
ЛК-4 + ЛК-4	Al	773	12	40
K-8 + K-8	Al	853	5	30

[1] For the copper interlayer, $p_{vac} = 1.3 \times 10^{-1}$ Pa. In all other cases, $p_{vac} = 0.1 \times 10^{-2}$ Pa.

can be made at all, because the red copper oxide is oxidized to black copper oxide. For this reason, the optimal vacuum for such cases is 1.33-0.133 Pa.

The mismatch in thermal expansion between the interlayer and the glass is responsible for the residual stresses that appear on cooling in the seal and may cause it to fail if their level exceeds the safe limit. Tensile stresses are especially dangerous, because glasses stand up well to compression. The level of residual stresses depends on the thickness of the metal part, the relaxation capacity of the materials involved, and the rate of cooling.

The vacuum diffusion bonding conditions for several Soviet-made grades of glass are listed in Table 15-8.

Several joint designs can be used for glass-metal seals (Fig. 15-2).

Butt type (Fig. 15-2*a*). In this type, the mating parts are in contact over the entire area. The seal performance is the best when the metal piece, *1*, has the glass part, *2*, on one side and a compensating ring, *3*, on the other. When the metal part matches the glass part in thermal expansion, this is a matched seal. When there is a mismatch in thermal expansion, an unmatched seal is produced. In designing a seal,

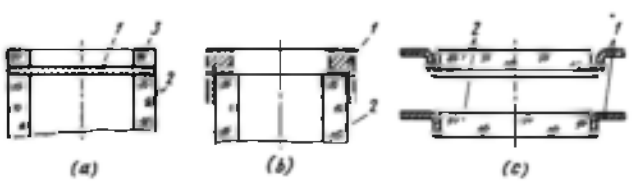

FIG. 15-2 Basic designs of glass-to-metal seals
(*a*) face-to-face; (*b*) compression (shrink-fit); (*c*) window-type

preference should be given to as thin a metal part as practicable. The reduction in thickness produces lower stresses that act on the metal and the glass.

Compression (or shrink) type (Fig. 15-2*b*). The metal part, *1*, is made in the form of a ferrule which encloses the glass part, *2*. The ferrule is made of a metal and with allowances such that, on cooling after bonding, it contracts to a shrink fit on the glass member. The thermal expansion coefficient of the metal member should be equal to or slightly greater than that of the glass. Otherwise, the glass part may be destroyed by excessive residual stresses.

Window type (Fig. 15-2*c*). This combines the features of the two previous designs. Where the glass, *2*, is to be sealed to a substantial metal member, *1*, the flanges should be fitted with bellows or membranes which will cancel the stresses arising due to changes in temperature.

Jigs and fixtures. These should provide for an accurate fit-up, avoid misalignment, uniformly transfer the bonding pressure to the mating surfaces, protect the glass against the likely thermal shocks on heating and cooling, and be inert towards the glass and the metal. These requirements are best met by graphite, especially a fine-porous grade. After machining, the graphite parts of a jig or fixture should be thoroughly washed in an ultrasonic bath, dried in an oven, and baked out in vacuum at 1 373 K for 30 min prior to their use.

Plant. Glass-metal seals can best be diffusion-bonded on units using radiation heating based on radiators fabricated from refractory and high-resistance alloys. With such heating elements, both heating and cooling can be maintained at a readily controllable, steady rate. Use may also be made of induction heating. In this case, a thin-walled cylinder must be put on the workpieces so that they can be heated by r.f. current. This type of heaters has a very low efficiency, and a thermal shock is likely to occur on heating.

The quality of seals can be tested by the usual methods applicable to all of diffusion bonding. Additionally, a polariscope-polarimeter can be used to determine the magnitude of residual stresses and inspect the seals for likely internal flaws, cracks, etc., which can be readily detected because of the transparency of glass.

15.3 Bonding of Ceramics to Metals

Ceramics refer to materials containing clay or other mineral powders, fired for strength at high temperature. In fact, the same production process applies to cermets, ferrites, carbides, nitrides, silicides and other non-clay materials. Some ceramic materials fuse at 2273-3213 K; their thermal conductivity is appreciably lower than that of metals. Ceramic materials have a high resistivity and dielectric strength. Owing to high mechanical strength, stability, and dimensional accuracy, ceramic components can be used at temperatures up to 1673-1973 K. The good chemical stability of ceramic and cermet components makes them indispensable in the chemical process industry. A major consumer of cermet products is the electronics industry.

A ceramic material contains both crystalline and vitreous constituents. Accordingly, all ceramic materials may be classed into those with a vitreous matrix (with

TABLE 15-9

Ceramic material	Designation	Principal oxides in the material
Magnesia-silicate:		
steatite	ВК-92, К-1, С-4А, С-14	SiO_2, MgO, BaO
forsterite	Ф-17, ЛФ-11, КВФ-4	MgO, SiO_2
Alumina-silica	102	Al_2O_3, SiO_2, BaO, CaO
High-alumina	22X, 22XC, A995, ВГ-IV, ГМ, М7, Sapphirite, Polycor	Al_2O_3
Berylla	Brokerite-9	BeO

the crystalline phase statistically dispersed in it) and those with a continuous crystalline phase which forms the skeleton of the ceramic body.

There is a great variety of structural types of ceramic materials, depending on chemical composition and the manner of manufacture. For example, vacuum-tight ceramic materials may have a vitreous or a crystalline matrix. The vitreous matrix occurs when the vitreous phase is not crystallized, partly crystallized, or completely crystallized, and the crystalline phase may consist of one, two or more crystalline compounds (this applies to ceramic-102 and steatites). A crystalline matrix occurs in a polyphase form within a limited bulk of glass and in a monophase, nonporous form (such as in Sapphirite 22XC, 22X, M-7, Ф-17, Polycor, and ГМ). The classification of Soviet-made vacuum-tight ceramic materials based on their composition is given in Fig. 15-9.

PROPERTIES OF ALUMINA-BASED CERAMICS. Several grades of alumina-based ceramic materials are available commercially for vacuum-tight seals. They contain a large proportion of SiO_2, BaO and CaO, with alumina accounting for 50-60% of the total. High-alumina ceramic materials contain over 80% of Al_2O_3. The chemical composition and principal physico-engineering properties of some alumina-based ceramic materials are listed in Tables 15-10 and 15-11.

TABLE 15-10

Material	Al_2O_3	SiO_2	Cr_2O_3	MnO	CaO	MgO	B_2O_3
22XC	94.4	2.76	0.49	2.35	—	—	—
M7	94.2	3.7	—	—	2.1	—	—
ВГ-IV	95.3	3.3	—	—	0.2	1.2	—
Sapphirite-16	98.0	—	—	—	—	0.5	1.5
Polycor	99.7	—	—	—	—	0.3	—

TABLE 15-11

Material	Bulk weight, kg m^{-3}	Static bending strength, MPa	Young's modulus, E, $\times 10^5$ MPa
22XC	3650	3440	3.08
M7	3600	2940	—
ВГ-IV	3650	3038	—
Sapphirite-16	3890	2940	3.23-4.31
Polycor	3980	2450	3.84

The value of ceramic materials lies primarily in their excellent thermal properties owing to which ceramic and cermet components and assemblies can reliably operate under isothermal conditions, exposed to sudden changes in temperature, and at large temperature gradients. Nevertheless thermal expansion, thermal conductivity, and specific heat have an appreciable effect on the strength of cermet components. The magnitude of thermal stresses arising in a cermet seal is primarily determined by the thermal expansion coefficient of the mating components. For the alumina-based materials in question, the average thermal expansion coefficient in the temperature range 293-1173 K is 8.0×10^{-6}-8.6×10^{-6} K^{-1}. For comparison, the thermal expansion coefficient for copper is 1.97×10^{-5} K^{-1} and for nickel, 1.63×10^{-5} K^{-1}.

In ceramic materials, the number of free electrons is small, so heat is conducted mainly by the elastic vibrations of the lattice. Because of this, the thermal conductivity of ceramic materials is one or two orders of magnitude lower than it is for metals. As with metals, an increase in temperature brings about a decrease in the thermal conductivity of ceramics. In contrast, an increase in temperature leads to a rise in the specific heat of most ceramic materials, irrespective of the lattice structure and constituents of the material. In the temperature range 373-1473 K, the specific heat of ceramic materials increases from 838 J kg^{-1} K^{-1} to 1 257 J kg^{-1} K^{-1}. The resistivity of ceramics decreases with rising temperature. As already noted, ceramic materials have a high dielectric strength. The same is true of compressive strength for densely sintered ceramics. For this reason ceramic components must be designed to operate in compression. The strength of a ceramic component depends on its size, and decreases with increasing diameter. The modulus of elasticity of ceramic materials depends on their chemical composition, temperature, and porosity.

DESIGNS OF CERAMIC-METAL SEALS. The difference in thermal expansion, thermal conductivity and heat transfer at a ceramic-to-metal seal gives rise to thermal stresses which may cause the component to fail. In designing a ceramic-to-metal seal, it is therefore important to take measures that will prevent the generation of thermal stresses.

In terms of thermal expansion, ceramic-to-metal seals may be classed into matched and unmatched. In terms of geometry and relative position of the components,

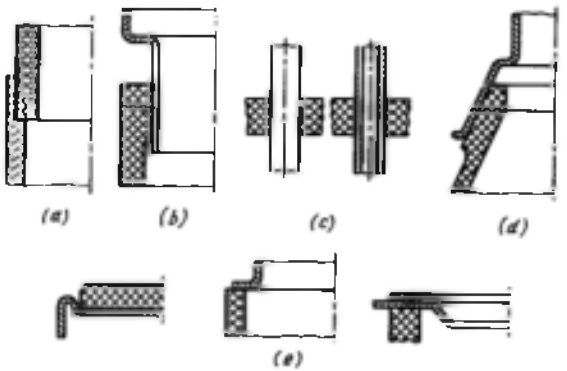

FIG. 15-3 Designs of ceramic-to-metal seals
(*a*) cylindrical compression; (*b*) and (*c*) cylindrical expansion; (*d*) taper compression; (*e*) face-to-face

the designs of ceramic-to-metal seals may be classed into the taper and cylindrical compression (or shrink-fit) types (the metal encloses the ceramic part), the cylindrical expansion-fit type (the ceramic part surrounds the metal part), and the face type (the metal and the ceramic establish contact over the face area). Examples of ceramic-to-metal seal designs are shown in Fig. 15-3.

PROCEDURES FOR THE DIFFUSION BONDING OF CERAMIC-TO-METAL SEALS. Diffusion bonding is mainly applied to the face type of ceramic-to-metal seals. The actual procedure is as follows. The components to be bonded together are machined at the joint. The metal part is machined to a roughness of 1.6 μm . After machining the parts are annealed to relieve stresses and baked out to remove dissolved and absorbed gases. Niobium, titanium, and tantalum are annealed (baked out) at a vacuum 1.3×10^{-2} Pa, whereas copper, Kovar, Fe-Ni alloy are treated in an atmosphere of dry hydrogen. Cermet parts are etched for surface cleaning and degreased with acetone or alcohol prior to fit-up. The joint sides on ceramic parts must be ground.

After surface preparation, the assembly is put into a bonding chamber where the work is raised to 1023-1573 K. At the specified temperature, the assembly is subjected to a bonding pressure of 4.90-19.6 MPa. Depending on the materials involved, the duration of pressure can be anywhere from 2 to 60 min. At the end of the holding time, the temperature is brought down to 473-523 K when the pressing load is removed. The key variables of diffusion bonding (see Table 15-12) are bonding temperature, bonding pressure, holding time, and the atmosphere in which the operation is carried out (this may be vacuum, hydrogen, or formier gas in the proportion $N_2/H_2 = 2 : 1$).

The atmosphere in which diffusion bonding is performed has a marked effect on the quality of the seal. Most high-alumina ceramic materials make vacuum-tight seals to copper in hydrogen and formier gas.

Diffusion bonding is effective in making good seals to ductile metals, such as copper and nickel. Fairly good seals have been made to titanium, niobium, Kovar,

TABLE 15-12

Atmosphere and materials	Bonding variables: Vacuum, Pa	Temperature, K	Pressure, $\times 10^7$ Pa	Holding time, min	UTS, σ_t, $\times 10^{-5}$ Pa
Hydrogen:					
22XC + copper	—	1273	1.76	10	1960
22XC + Kovar	—	1473	1.76	10	980
102 + 12X18H10T steel	—	1473	1.76	15	1127
102 ceramic + Kovar	—	1473	1.76	10	980
102 ceramic + copper	—	1273	1.76	10	1176
M7 + copper	—	1273	1.76	10	1176
MГ + copper	—	1273	1.76	10	1176
Vacuum:					
22XC + titanium	1×10^{-1}	1273	1.17	10	1176
22XC + copper	1×10^{-1}	1273	1.96	15	—
ЦТС-19 + AMr6 alloy	5×10^{-2}	793	0.88	40	98
ЦТС-19 + M1 + 12X18H10T steel	5×10^{-2}	1113	1.96	40	137
ЦТС-19 + copper	5×10^{-2}	1013	0.78	50	147
M7 + copper	1×10^{-1}	1273	1.76	40	2058 (bend)
ГМ + titanium	1×10^{-2}	1423	1.37	5	1225 (bend)
Sapphire + 46H alloy	5×10^{-2}	1523	0.29	10	—
ZnS + Kovar	1×10^{-1}	1123	245	20	—

12X18H10T steel, and Nichrome. Direct seals to refractory metals are never vacuum-tight. Therefore, they are made with an interlayer of copper, nickel and titanium, or to a metal-plated ceramic surface.

Diffusion-bonded ceramic-to-metal seals offer a number of abvantages over brazed ones. Among other things, the seals can stand up to higher temperatures in service, endure repeated heating in vacuum without a loss of vacuum tightness (up to 15 thermal shocks in the 293K-873K-293K cycle), have a strength which is half as much again as that of brazed seals, and possess better insulating properties.

BONDING OF OPTICAL CERAMIC MATERIALS TO METALS. Optical ceramic materials are a class of artificial polycrystalline hot-pressed materials transparent to visible and infra-red light. They are single-phase ceramics based on the fluorides of alkali-earth metals, chalcogenides of zinc and cadmium, and some oxides. In optical properties they are similar to single crystals, but they are superior to the latter in thermal and mechanical properties. In terms of physical properties, they stand midway between semiconductors and dielectrics. They go to make lenses, windows, filters and some other products. Owing to the rigidly oriented covalent bonds in the lattice, optical ceramic materials are brittle at room temperature, but they become capable of plastic deformation when heated to 973-1173 K. Beyond the brittle threshold, they deform by the creep mechanism at a fairly high rate. This feature is

responsible for the fact that the actual contact between the mating surfaces in the diffusion bonding of an optical ceramic material to a metal is established very soon and the surfaces are readily activated.

Most detailed data have been obtained on the bondability of metals to optical ceramic materials based on zinc sulphide, ZnS. The relationships thus deduced can to a certain extent be applied to other optical ceramic materials. A distinction of the bonding process is that it must be carried out in an inert gas atmosphere rather than in vacuum, because in the latter case the metal and the optical ceramic material react at the bonding temperature to form gaseous products which tend to etch out the two materials.

Vacuum-tight seals of zinc sulphide to copper and Kovar can successfully be made in an argon atmosphere at $T = 1\,123$ K, $p_b = 784$-980 MPa, and $t = 40$ min. These metals are chosen because the stresses due to differential thermal expansion can readily be minimized. The relatively low level of stresses in ceramic-to-Kovar seals arises from the fact that the two materials are a nearly perfect match in terms of thermal expansion at temperatures below the Curie point of Kovar. In the case of seals to copper, the stresses are effectively relaxed owing to the plastic deformation of the thin copper collar (not over 0.5 mm thick).

No direct reactions (of the redox type) can occur between the ZnS part and the metal part of a seal under the bonding conditions, because zinc has a higher affinity for sulphur at any temperature. A diffusion-bonded seal is produced owing to the dissolution of the ceramic material with the formation of solid solutions of zinc and sulphur in the seal. The equilibrium concentration of solid solutions is 7% in sealing to copper, and 2% in sealing to Kovar at the bonding temperature. This low concentration of solid solution seems to be insufficient for the formation of a transition layer which would provide for a gradual change in physico-chemical properties from the ceramic material to the metal. The strength of the joints made as described above is 80-90% that of the ceramic material.

One way to improve the joint strength is to sulphidize the metal surface prior to bonding. In the experiments involving the sealing of ZnS to presulphidized specimens of copper, iron and Kovar it has been found that the joints thus made match the ceramic material in strength and that failure occurs in the ceramic. Given the same bonding temperature, sulphidizing makes it possible almost to halve the holding time (the duration of bonding pressure).

From an engineering and economic point of view, it is preferable to make some designs of seals, such as optical windows, by a hot-pressing technique in which the ceramic material is fabricated and bonded to the metal at the same time. When the ZnS powder and the metal are heated, the metal takes on a sulphide film which is formed from the free sulphur and hydrogen sulphide present in the ZnS powder owing to the manner in which it is prepared. This combination of two steps in a single operation avoids the use of a separate sulphidizing step. Nor is it necessary to prepare the ceramic component for bonding. Since, however, this technique involves high pressing loads, the use of copper for seals is out of the question. Instead, the metal component is fabricated from Kovar. The hot pressing load is by an order of magnitude higher than the pressure used in conventional diffusion bonding, and

it might cause an excessive distortion in the metal element of the window. Fortunately, with the operation conducted in a compression mould, the metal is in compression from nearly all sides. Therefore, the thickness of the metal collar is reduced by not more than the allowed 10-15%. When the compression mould is being put together, the metal collar (mount) is placed in a cavity milled to conform precisely to its contour. Then the mould is filled with a metered quantity of ZnS powder, and is placed in a vacuum chamber where the powder is cold-pressed under a low pressure (1.96×10^7 Pa) in order to compact the powder a little. Following that, the work is hot-pressed at a temperature of 1 123 K, a bonding pressure of 245 MPa, and a vacuum of 1×10^{-1} Pa, with the diffusion treatment maintained for 20 min. To minimize the stresses arising from the difference in thermal expansion between the ceramic and the metal (Kovar), the cooling rate is maintained at 5-7 K min^{-1} down to a temperature of 773 K, then at 10-15 K min^{-1}. With this procedure, window seals of the face and compression (shrink fit) type can be made in a single cycle. In a trial, windows with a compression seal retained vacuum tightness after 20 thermal cycles, and windows with a face-type seal, 4 or 5 thermal cycles.

15.4 Bonding of Semiconductors to Metals

Semiconductors differ qualitatively from metals in the nature of chemical bonds, structure and physico-mechanical properties. Their difference from dielectrics is one of a quantitative character.

At room temperature, the conductivity of semiconductors ranges between 10^{-7} and $10^5\ \Omega^{-1}\ m^{-1}$, depending on the kind and amount of impurities present, the lattice structure, and ambient conditions (temperature, pressure, presence of electric and magnetic fields, illumination and exposure to nuclear radiation). In accord with the band theory of solids, valence electrons in metals can readily move to levels in the conduction band and contribute to the flow of electric current. In semiconductors, the valence band is completely filled and separated from the conduction band by a forbidden band or gap. The forbidden band of semiconductors is $0.16 \times 10^{-19} - 5.1 \times 10^{-19}$ J wide. Solids with a wider forbidden band belong to dielectrics.

A semiconductor device is basically a crystal of a semiconducting material with one or several electron-hole (or p-n) junctions, produced by doping various areas in the crystal with appropriate impurities.

SIMPLE SEMICONDUCTORS. These include twelve elements: boron, carbon, silicon, phosphorus, sulphur, selenium, germanium, arsenic, gray tin, antimony, tellurium, and iodine. A generalized index of the nature of chemical bonding in this subgroup is the ordinal number of an element in the Periodic Table. Simple semiconductors have covalent bonds which are formed by the interaction of two electrons with opposite spin.

BINARY SEMICONDUCTORS. These fall in a common class of compounds having the same stoichiometric relation and frequently consisting of elements belonging to the same group in the Periodic Table. Symbolically, they are designated $A^n B^{8-n}$, where A is an element in Group n, and B, an element in Group $(8 - n)$.

A generalized index of the nature of chemical bonding in this class of compounds is the average atomic number of the compound. These binary compounds show mixed covalent-ionic-metallic bonds, with each atom of one kind surrounded by four nearest neighbouring atoms of the other kind to form a tetrahedral structure. Such semiconductors crystallize into a sphalerite or wurzite structure. At present, the leading binary semiconductors are those answering the general formula $A^{III}B^{V}$, such as InSb, GaAs, GaP, AlN, BN, and some others. They are widely used in the manufacture of r.f. and high-temperature transistors, tunnel diodes photocells, lasers, microwave oscillators, and similar devices.

BASIC PROPERTIES OF SEMICONDUCTORS. Semiconductor crystals have the lattice structure of diamond and display spatial symmetry. Their density ranges between 2 300 and 8 200 kg m^{-3}. As the atomic number increases, the bond energy goes down, the forbidden gap narrows, and the metallic component of the bond is increased. Data on some of the semiconductor crystals are listed in Table 15-13.

Of all semiconductors, silicon has the lowest coefficient of thermal expansion. As the temperature is raised, the thermal expansion coefficient increases (Fig. 15-4). Also, it depends on the crystallographic plane; for example, it is a maximum in the ⟨100⟩ plane and a minimum in the ⟨111⟩ plane for silicon and germanium. In most semiconductors, heat is transported by phonons, the contribution of electrons being very small. The thermal conductivity of semiconductors decreases with increasing temperature. It is also affected by the degree of doping, lattice structure, and number of defects. The resistivity of semiconductors decreases with increasing temperature, and does so exponentially rather than linearly (as is the case with metals):

$$\rho = A \exp(\Delta E/kT) \tag{15-1}$$

where ΔE is the width of the forbidden band, $k = 1.38 \times 10^{-23}$ J K^{-1} is Boltzmann's constant, T is the absolute temperature, and A is a coefficient depending on the nature of the semiconductor.

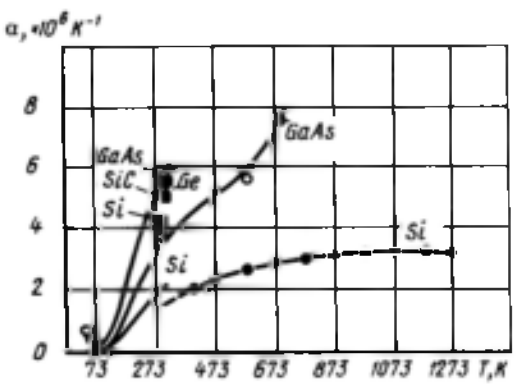

FIG. 15-4 Thermal expansion coefficient of semiconductors as a function of temperature

TABLE 15-13

Properties	Semiconductors			
	Si	Ge	GaAs	SiC
Group in Periodic Table	IVB	IVB	$A^{III}B^{V}$	$A^{IV}B^{IV}$
Atomic No.	14	32	32	10
Relative atomic mass	28.69	72.59	144.63	40.10
Lattice type	fcc, diamond type	fcc, diamond type	Sphalerite	Sphalerite
Lattice period, a, $\times 10^{-10}$ m	5.43	5.66	5.65	4.35
Density, γ, kg m^{-3}	2330	5320	5350	3110
Fusion point, T, K	1683	1213	1511	3073
Forbidden band width, ΔE, $\times 10^{-19}$ J, at 300 K	1.73	1.06	2.21	5.02
Electron mobility, μ, m^{2}V^{-1} s^{-1}, at 293 K	0.155	0.420	0.790	0.0032
Thermal expansion coefficient, α, $\times 10^{6}$ K^{-1}, at 300 K	2.35	5.75	5.84	5.10
Debye temperature, Θ_D, K	680	406	355	—
Work function, Φ, $\times 10^{-19}$ J	7.84	7.52	7.50	11.34
Thermal conductivity, κ, g m^{-1} K^{-1}	109.0	53.7	155.0	8.38
Specific heat, C_p, J kg^{-1} K^{-1}	1627	1914	1918	2198
Latent heat of fusion, Q_f, $\times 10^{5}$ J kg^{-1}	42.45	28.35	—	—
Resistivity, ρ, $\times 10^{6}$ Ω m	0.23	0.51	—	10.0
Poisson's ratio, σ	0.28	0.21	0.29	—
Mohs' scratch number	6.5	6.2	6.7	9.6
Microhardness, H_μ, $\times 10^{7}$ Pa	1127-1303	764-828	720-750	2822-3106
Young's modulus, E, $\times 10^{7}$ MPa	16.4	13.4	11.1	14.2

The resistance of semiconductors can be varied by exposing them to light, radiation, plastic deformation, or other external factors, or by varying their chemical composition (by dissolving the impurities).

The yield strength of semiconductors varies with temperature exponentially. At room temperature, germanium and silicon can be cleaved without an appreciable plastic deformation (compression at a stress of 157 MPa and 245 MPa, respectively). For gallium arsenide and silicon carbide the respective figures are 176 MPa and 196 MPa. In diamond-type semiconductors, ductility comparable with that of metals can only be achieved near the fusion point. In static tension, this occurs around 723 K for germanium, 923 K for silicon, and 673 K for gallium arsenide. In bending, this occurs at temperatures by 75-100 K lower, whereas in creep tests this

happens at still lower temperatures. The mechanical strength of semiconductors decreases with the decrease in the strength of chemical bonds.

DESIGN OF SEMICONDUCTOR-METAL CONTACT. When a semiconductor and a metal are brought together, a rectifying contact or an ohmic contact can be established. With a rectifying contact, a charge-depleted potential barrier is formed at the contact. With an ohmic contact, the region at the interface has a high concentration of majority carriers. The properties of the rectifying contact are widely used in thin-film structures (thin-film diodes and transistors). The ohmic contact is utilized in diodes, transistors, integrated circuits, and some other cases. It has a very low resistance, the potential difference across the junction is proportional to the current flowing through it, the waveform passing across the contact remains undistorted, and the associated noise level is low.

The principal form of joint for semiconductor-metal devices is the face type. The most commonly used designs are shown in Fig. 15-5.

The metals and alloys used to make an ohmic contact with semiconductors must meet the following requirements:

— they should increase the concentration of majority carriers in the semiconductor or be neutral in that respect;

— they should be mutually soluble with the semiconductor and have a fusion point which is below that of the semiconductor (so as to form a low-melting-point eutectic with the semiconductor);

— they should have high thermal and electric conductivity, match the semiconductor in thermal expansion very closely over a wide temperature range, possess sufficient ductility so as to avoid cracking and internal stresses in the wake of temperature variations. For metals with a marked expansion mismatch, use should be made of compensating interlayers closely matching the semiconductor in thermal expansion;

— they should only slowly diffuse into the semiconductor at the bonding temperature and in service;

— no phase transitions should occur over the temperature range of contact formation;

— they should perform equally well under conditions of varying temperature, static and dynamic loads;

— they should meet the requirements specified for the joint itself.

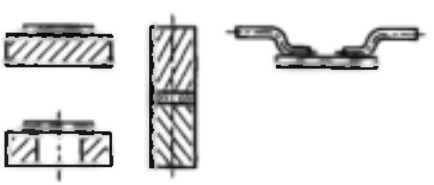

FIG. 15-5 Designs of semiconductor-to-metal bonded joints

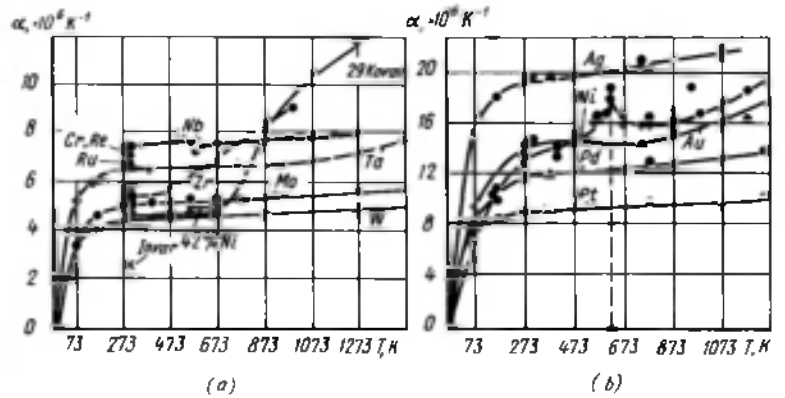

FIG. 15-6 Thermal expansion coefficient of metals (*a*) and alloys (*b*) as a function of temperature

Silver, gold, nickel and aluminium are ductile metals with a face-centred cubic (fcc) structure. They have very high thermal and electric conductivity, low solubility in solid germanium, silicon and gallium arsenide. Silver, aluminium and gold form with germanium and silicon low-melting-point eutectics. Gold diffuses at a medium rate into germanium, rapidly into silicon, and slowly into gallium arsenide. Nickel is a rapidly diffusing impurity which shortens the lifetime of minority carriers. Silver, gold and nickel are highly corrosion-resistant. In the fabrication of semiconductor-metal junctions by diffusion bonding, they are used (Fig. 15-6*b*) as backers, interlayers, and foil 5-20 μm thick.

The thermal stresses that may arise during the bonding process can be minimized by closely matching the semiconductor and the metal in terms of thermal expansion and thermal conductivity. The metals that come very closely to semiconductors in terms of thermal expansion (Fig. 15-6*a*) are tungsten, molybdenum, chromium and tantalum (6.6×10^{-6} K^{-1}), niobium (7.2×10^{-6} K^{-1}) and some others. They have a body-centred cubic (bcc) structure which is not so closely packed. The fusion point of these metals ranges from 2 148 K for chromium to 3 683 K for tungsten, which is 1.1-2.7 times the fusion point of the semiconductors concerned. These metals have a high energy of activation (from 3.7×10^7 to 4.2×10^7 J kg^{-1}) and large coefficients of self-diffusion (from 2×10^{-4} to 1.6×10^{-3} m^2 s^{-1}), for which reason the diffusion bonding of semiconductors to metals calls for greater energy input. All of these metals are mechanically strong, have a high resistivity, and stand up well to corrosion.

Of alloys, those matching semiconductors in thermal expansion most closely are Kovar, Fe-Ni alloys, chromium steels, and Nichroms (20% Cr-80% Ni and 15% Cr-60% Ni). Kovar has a low thermal conductivity, a high resistivity, and good corrosion resistance.

The metals and alloys listed above are used as diffusion-bonded cases for semiconductor devices and thermal compensators 0.5-1 mm thick.

PROCEDURES FOR THE DIFFUSION BONDING OF SEMICONDUCTORS AND METALS. Preparatory steps include the slicing of semiconductor ingots prepared by known processes into wafers up to 100 mm in diameter and 0.2-10 mm thick (thinner wafers would readily break). The final step is the polishing of wafers to a roughness of 0.1-0.05 μm, so the disturbed layer is not more than 0.2-1 μm thick and the surface is free from defects. After machining, wafers are treated for removal of organic, ionic and metallic contaminants in various substances and allowed to dry. Following that, *p-n* junctions are produced and components are assembled into devices (preferably by diffusion bonding). Metal parts are fabricated by stamping and cutting. They are machined to a surface roughness of 1.25-0.63 μm and polished flat to 0.01-0.02 μm.

Prior to diffusion bonding, semiconductor components made of silicon, germanium, gallium arsenide and silicon carbide are degreased with carbon tetrachloride and alcohol. Metal parts made of molybdenum, tungsten and Kovar are degreased in alkaline solutions and pickled in various mixtures of acids.

The diffusion bonding of simple semiconductors (silicon and germanium) is both similar and different from that of binary semiconductors (gallium arsenide and silicon carbide). The differences arise from the very nature, structure and physicomechanical properties of the materials.

Each group of semiconductors can be diffusion-bonded either to unplated or to plated metals. In the former case, the bonding temperature is an exponential function of bonding pressure (curve *I* in Fig. 15-7), so as the bonding temperature is raised, the bonding pressure must be brought down. The pressure is minimal in the case of diffusion brazing, and drops to zero when a metal is diffusion-fused into a semiconductor. Conversely, as the bonding temperature is reduced, the bonding pressure must be raised. This relation between temperature and pressure exists because the diffusion bond is now formed primarily by plastic deformation. The limiting case is when the bonding temperature is less than half the melting point of the semiconductor — now no diffusion bond will be formed even at a bonding pressure of as high as 98-196 MPa.

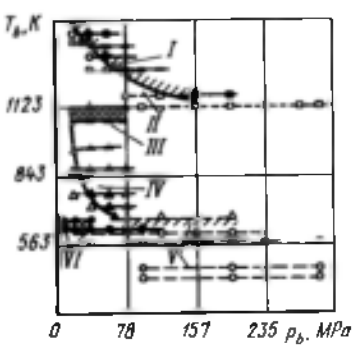

FIG. 15-7 Bonding temperature as a function of bonding pressure for Si + Mo, Si + silver-plated Mo, and Si + gold-plated Mo

The following empirical relation has been established experimentally between bonding temperature, T_b, and bonding pressure, p_b, for a silicon-to-molybdenum joint (unplated surfaces)

$$T_b = 1156 \exp(-0.017 p_b) \qquad (15\text{-}2)$$

As has been found, no diffusion bond can be formed between unplated wafers of silicon, germanium, gallium arsenide and silicon carbide and a molybdenum specimen at a temperature of 1 073 K, 823 K, 873 K and 1 173 K, respectively, a bonding pressure of 196 MPa, a holding time of up to 60 min, a vacuum of 1×10^{-2} Pa (region *II* in Fig. 15-7). Silicon can be diffusion-bonded to tungsten (which matches silicon very closely in thermal expansion) at a temperature of 1373-1473 K and a bonding pressure of 29.4-39.2 MPa. Failure of a diffusion bond to form at a bonding temperature less than 0.5 or 0.6 of the melting point of the semiconductor stems from the lack of energy required to build up the oscillation amplitude of atoms, the atoms of the refractory metals cannot properly interact with those of the semiconductors, and the diffusion rate is low.

When semiconductors are diffusion-bonded to gold-, silver- or nickel-plated molybdenum specimens, the bonding temperature is a hyperbolic function of pressure (curve *IV* in Fig. 15-7). This is an indication that the bond gains in strength primarily due to the mutual diffusion between the rapidly diffusing plating and the diamond-like semiconductor. With nickel-plated molybdenum, the bonding temperature can be brought down by 300 K for silicon and germanium, by 450 K for gallium arsenide (curve *1* in Fig. 15-8), and by 400 K for silicon carbide, the bonding pressure being held at the same value of 39.2 MPa. The lower asymptotes of the hyperbolic curves lie at a temperature of 673 K for silicon, germanium and gallium arsenide, and at a temperature of 832 K for silicon carbides. Because of this, no diffusion bond can be produced at a bonding temperature which is less than 0.3 or 0.4 of the melting point of the semiconductor, even when a rapidly diffusing plating material, such as nickel, is used. With silver-plated molybdenum, the bonding

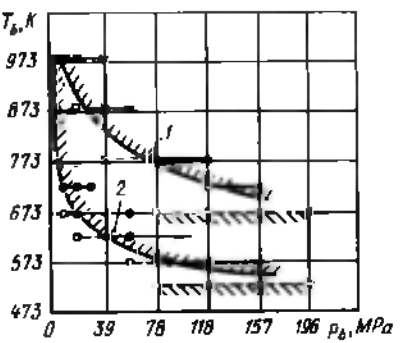

FIG. 15-8 Bonding temperature as a function of bonding pressure for GaAs + nickel-plated Mo and GaAs + nickel-plated Kovar

temperature can be reduced by a factor of 0.2-1.4, so it is 773 K for silicon and 973 K for silicon carbide, while keeping the bonding pressure unchanged, or the bonding pressure can be halved or even reduced to one-third (19.6-9.8 MPa). Silicon and germanium cannot be diffusion-bonded to silver-plated molybdenum at temperatures higher than 1 103 K and 924 K, respectively, because of the formation of eutectics at the contact (region *III* in Fig. 15-7). For the diffusion bonding of unplated silicon to silver-plated molybdenum, the empirical relation between T_b and p_b has the form

$$T_b = 1\,052 p^{-0.54} \qquad (15\text{-}3)$$

Gold plating makes it possible to bring down the bonding pressure to as low as 0.98-4.90 MPa and the bonding temperature to 646 K for silicon (region *VI* in Fig. 15-7), to 629 K for germanium, and to 873 K for silicon carbide. At the temperatures stated, silicon and germanium form eutectics with gold, so the joint is actually produced by diffusion brazing in vacuum. When the bonding temperature is brought down from 623 K to 423 K and the bonding pressure is raised to 294 MPa (region *V* in Fig. 15-7), a diffusion bond is produced, but the adhesion area in the joint accounts for only 20% to 90% of the total contact area, the dislocation density is increased 6-8 times, and heavier mechanical stresses are generated, leading to microcracking and phase transitions at the *p-n* junction. No eutectics are formed by silicon carbide with silver or gold.

The limiting temperature at which no bond is formed between silicon, germanium and silicon carbide, on the one hand, and gold, silver and nickel plating, on the other, is 623-673 K. For gallium arsenide and nickel plating the limiting temperature is 773-873 K. The diffusion bonding of silicon and germanium to aluminium-plated molybdenum and tungsten should be done below the eutectic temperature which is 773-873 K for silicon and 643-673 K for germanium.

In the diffusion bonding of semiconductors to metals the bonding pressure (uniaxial compression) brings about elastic deformation accompanied by a continuous motion and multiplication of dislocations which are oriented most favourably relative to the applied stress. In diamond-like crystals, shear mainly occurs in an octahedral plane, forming slip bands. To minimize the difference in the deformation of crystals, the pressing load should not produce a degree of deformation in excess of 3% or 4%, and it should be held constant until the joint has cooled completely. Studies have shown that the mobility of point defects produced in diamond-type crystals by plastic deformation is substantially lower than their mobility caused by heating and cooling. For a strong diffusion-bonded joint to be made between a semiconductor and a metal or alloy, the rate of cooling must be set at half the value adopted for heating (Table 15-14).

According to microstructural analysis, the transition region in a diffusion-bonded joint between silicon and unplated molybdenum or tungsten consists of molybdenum silicide ($MoSi_2$) or tungsten silicide (WSi_2), and solid solutions of silicon in molybdenum or tungsten. In joints involving silicon carbide, the transition region additionally contains molybdenum carbide which doubles the microhardness

TABLE 15-14

Semiconductor	Rate of heating, K min^{-1}	Rate of cooling, K min^{-1}
Silicon	50-60	20-30
Germanium	40-50	20-30
Gallium arsenide	20-30	10-20
Silicon carbide	30-40	10-20

of the transition region. In the case of bonding silicon and silicon carbide to silver- or gold-plated molybdenum, the microhardness of the transition region is increased by 20-30%, which is an indication that these metals dissolve poorly in solid semiconductors.

The manufacture of semiconductor devices widely uses Fe-Ni-Co alloys (such as Kovar) which match semiconductors in thermal expansion almost completely. The hyperbolic curves which separate the regions in which semiconductors can or cannot be diffusion-bonded to nickel-plated Kovar run more steeply than those for nickel-plated molybdenum (curve *2* in Fig. 15-8). This is an indication that a metal with a lower melting point and a nickel plating has a sufficient thermal activity at the low temperatures of vacuum diffusion bonding (T_b is 20% of the melting point of the semiconductor), its atoms can readily overcome the potential barrier between the lattice sites in silicon carbide and gallium arsenide, and a monolithic diffusion-bonded joint is formed. Nickel-plating on Kovar makes it possible to bring the bonding temperature down to 873 K in the case of silicon carbide and to 673 K in the case of gallium arsenide, and to reduce the bonding pressure by a factor of 2.5-4 for silicon carbide and by a factor of 10-12 for gallium arsenide.

As the bonding temperature is brought down, the duration of diffusion treatment, t_b, is increased exponentially. The empirical relation deduced for the diffusion bonding of silicon to unplated molybdenum (curve *I* in Fig. 15-9) has the form

$$T_b = 1211 \exp(-0.012 t_b) \tag{15-4}$$

For silicon and silicon carbide, the relation has the same form (curves *I* and *IV* in Fig. 15-9), and that for germanium (curve *III* in Fig. 15-9) is the same as curve *I*. This is an indication of an identical behaviour of semiconductors having a diamond-type lattice. When molybdenum is nickel-, silver- or gold-plated (curve *VI* in Fig. 15-9), the duration of diffusion treatment can be cut down by a factor of 1.5-2, that is, reduced to 5-7 min. The curves for the relation $T_b = f(t)$ with nickel and silver plating run together (curve *IV* in Fig. 15-9). This is an indication that the two plating materials have an identical accelerating effect on diffusion processes in the joining of semiconductors to molybdenum. When the bonding temperature is reduced to below 1 123 K (for unplated surfaces) and 673 K (for plated surfaces) and the duration of diffusion treatment (holding time) is increased to 60 min (regions *II* and *V* in Fig. 15-9), no diffusion bond is formed.

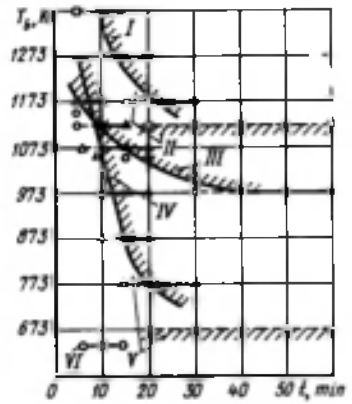

FIG. 15-9 Bonding temperature as a function of holding time for joints of Si and SiC to Mo, silver-, gold-, and nickel-plated Mo and Ge to Mo

The conditions for the diffusion bonding of semiconductors to metals and alloys are listed in Table 15-15.

Semiconductor devices fabricated with the aid of diffusion bonding have been subjected to all-round tests specified for a particular class of devices. It has been found that their volt-ampere characteristic is by 10-30% better than that of conventional devices.

TABLE 15-15

Materials joined	Plating	Bonding temperature, K	Bonding pressure, MPa	Holding time, min
Si + Mo	None	1273	68.6	15
Ge + Mo	None	973	29.4	15
GaAs + Mo	None	1073	58.8	20
SiC + Mo	None	1473	68.6	10
Si + W	None	1473	39.2	15
Si + Mo	Nickel	873	19.6	15
	Silver	873	19.6	15
	Gold	623	78.4	30
	Aluminium	773	19.6	20
Si + W	Aluminium	823	19.6	20
SiC + Mo	Nickel	1173	9.8	10
	Silver	1073	19.6	10
	Gold	973	9.8	10
GaAs + Mo	Nickel	973	9.8	15
GaAs + Kovar	Nickel	773	9.8	15
SiC + Mo	Nickel	1073	39.2	10
SiC + Kovar	Nickel	973	19.6	10
Ge + Mo	Nickel	773	19.6	15

Note. In all cases, the vacuum in the bonding chamber is 1×10^{-2} Pa.

As has been proved at the PNILDSV, diffusion bonding can make semiconductor-metal contacts with an area of up to 700 mm^2. The devices thus fabricated include silicon transistors and diodes, germanium transistors, GaAs photocathodes for photomultiplier tubes, pressure sensors based on silicon, gallium arsenide and silicon carbide, and a wide range of other devices.

15.5 Bonding of Ferrites to Metals

The term ferrites has come to mean a whole class of ceramic-like magnetic materials with Fe_2O_3 as a major component, and with certain oxides, such as oxides of zinc, nickel and manganese, as dopants.

The principal requirements that a ferrite-metal assembly should meet are mechanical strength, constancy of magnetic properties, and effective heat abstraction. At present, diffusion bonding is applied to a wide range of ferrites, such as yttrium-gadolinium garnet ferrites joined to copper; nickel-zinc and manganese-zinc ferrites joined with a glass interlayer; and nickel-manganese ferrites joined at a low temperature with an interlayer of a readily fusible metal.

DIFFUSION BONDING OF GARNET FERRITES. These are ferrites having the garnet crystal structure, most frequently used at microwave frequencies. Diffusion bonding is used to join garnet components to metal fixtures inside microwave devices. Diffusion-bonded joints offer a number of advantages, such as good heat abstraction, high stability towards thermal and dynamic loads, and serviceability up to 873-923 K. As studies have shown, the exposure to high temperature in the course of diffusion bonding does not affect the magnetic properties of the garnets. However, a change in these properties can be brought about by the formation of qualitatively new interaction products in the transition region. The magnitude of the change will depend on the chemical composition and size of the transition region.

Yttrium-gadolinium garnets are diffusion-bonded with the use of a copper interlayer 0.6 mm thick. The preparatory steps include boiling in distilled water for 3h (which may be replaced with ultrasonic cleaning) and annealing at 873 K in air for 15 min. Optimal results have been obtained at a bonding temperature of 1173-1223 K, a bonding pressure of 16-20 MPa, a holding time of 15-20 min and a vacuum of 0.133 Pa. The joints thus made have an ultimate tensile strength of 6000-6500 MPa. The transition region between the copper and the ferrite is 5-7 μm.

DIFFUSION BONDING OF FERRITES WITH A GLASS INTERLAYER. This technique is applied to Ni-Zn and Mn-Zn ferrites to form gaps in magnetic recording heads (for tape recorders, computers, and similar applications). Now two zones are formed in the transition region, namely a diffusion zone due to the diffusion of the ferrite components into the glass, and a metamorphic zone where the ferrite structure is changed owing to the diffusion and chemical interaction of the glass components with the ferrite.

Because of the presence of a transition region in ferrite-glass joints the effective gap width may exceed its physical width. To avoid this, diffusion bonding should preferably be conducted at T_b = 923-1023 K, p_b = 2-5 MPa, and t = 15-20 min.

The interlayer should be made of window glass 5-10 μm thick. An advantage of this design is an increase in the wear resistance of the gap edges. A disadvantage is that if the component is heated in vacuum for a long time and to a high temperature, the stoichiometric ratio of the ferrite may change. To avoid this, a low vacuum or an inert atmosphere should be used.

DIFFUSION BONDING OF FERRITES WITH READILY FUSIBLE INTERLAYERS. Some of the commercially used ferrites cannot be heated to high temperatures because of an impairment in their magnetic properties. In a number of cases the maximum temperature is 623 K. A way out for joining such ferrites to another ferrite or some other material is to use a fusible interlayer which forms a liquid phase at the time of bonding. This fusible interlayer can be produced, for example, by a consecutive vacuum evaporation of nickel, copper and indium onto the mating surfaces; indium may be replaced with tin or some other low-melting-point metal. Thus prepared, the components are assembled in a jig or fixture and the assembly is heated in vacuum until the most fusible metal melts. The excess of the liquid phase is expelled from the joint and fills the irregularities usually present on the mating surfaces. The duration of constant-temperature diffusion treatment is chosen to be sufficient for the enrichment of the liquid phase with the less fusible copper or other metal and the consequent formation of a binary or ternary system. As an example, ferrite specimens diffusion-bonded with a nickel-copper-indium interlayer, when held at 623 K for 40 min, had an ultimate tensile strength of 4 100 MPa, which is twice the strength of pure indium. The joints thus made can endure a temperature which is substantially higher than the fusion point of the most fusible component.

15.6 Bonding of Graphite to Metals

Graphite components are often included in metal structures and are used in combination with a wide range of metals.

A careful study of the known mechanisms for removal of oxides upon the heating of a metal in vacuum and a series of experiments have shown that at the temperatures and vacua ordinarily used for diffusion bonding the vaporization and dissociation of oxides play an insignificant role, if any. The situation can be changed if the metal is heated in the presence of graphite. Now the oxides can dissociate, because the carbon reacts with oxygen to form CO and CO_2, and the partial pressure of the oxygen present falls well below the equilibrium value. It is likely that such events occur only during the initial stage in the bonding of graphite to steels. Otherwise, they would entail an increase in the thickness of hard products on the graphite — a thing not actually observed.

The rate of reduction is a function of many factors. In addition to ambient conditions (temperature, pressure, nature of reduction), the reaction rate is affected by the physico-chemical properties of the material being reduced, its mineralogical composition, structure, surface quality, etc. So far, the cumulative effect of all the factors taken together has not been considered and no all-embracing mathematical relation has been developed.

In an experiment, graphite with a porosity of up to 80% was diffusion-bonded to BT1 titanium alloy. In order to make a full-strength bond, it was found necessary

TABLE 15-16

Materials joined	Plating on graphite	Bonding temperature, K	Bonding pressure, MPa	Holding time, min
BT1 titanium alloy + АГ-1500 graphite	Nickel	1123	0.98	35
BT1 titanium alloy + АГ-1500 graphite	None	1373	6.86	45
Ст 3 steel + АГ-1500 graphite	None	1273	9.80	30
20Х13 steel + АГ-1500 graphite	None	1273	9.80	30

Note. The vacuum in the bonding chamber, $p_{vac} = 1 \times 10^{-1}$ Pa.

to use a vacuum of 1×10^{-1} Pa and a bonding pressure of not over 4.9 MPa. However, this pressing load caused deformation on the titanium side. To avoid this, the pressing load was brought down to 2.9 MPa. Because of the organic binder present in the graphite, it could not be bonded to the titanium directly. In order to remove the binder, the specimens were placed in a solution of carbon tetrachloride prior to joining. When subjected to mechanical tests, the bonded specimens failed through the graphite. Metallographic analysis showed that the materials had completely merged together at the contact.

Suggested conditions for bonding graphite to metals are listed in Table 15-16.

On the basis of experimental data it may be concluded that in the diffusion bonding of graphite to metals the oxide coat is removed in a matter of seconds over a wide range of temperatures (1073-1273 K), bonding pressures (0.98-19.60 MPa), and vacuum (10^{-3} Pa). Therefore, this is not a limiting factor in making full-strength joints. Accordingly, there is no need for removal of oxides from the metal surfaces prior to bonding, and the rate of full-strength bond formation does not materially depend on the degree of vacuum.

Bibliography

1. KESAMANLY, F. P., NASLEDOV, D. N. (eds). *Gallium Arsenide — Preparation, Properties and Applications*. Nauka, Moscow, 1973 (in Russian).
2. BATYGUIN, V. N., METELKIN, I. I., RESHETNIKOV, A. M. *Vacuum-Tight Ceramics and Ceramic-to-Metal Seals*. Energiya, Moscow, 1973 (in Russian).
3. BOLTAKS, B. I. *Diffusion in Semiconductors*. Phizmatguiz, Moscow, 1961 (in Russian).
4. *Diffusion Bonding of Silicon to Metals*. In: "Vacuum Diffusion Bonding of Metals, Alloys and Nonmetals", edited by N. F. KAZAKOV. PNILDSV, Moscow, 1973, pp. 139-146 (in Russian).
5. KAZAKOV, N. F. *Diffusion Bonding of Materials*. Mashinostroyeniye, Moscow, 1976 (in Russian).
6. PRESNOV, V. A. (ed.). *Ceramics and Ceramic-to-Metal Seals in Engineering*. Atomizdat, Moscow, 1969 (in Russian).

7. KIREEV, P. S. *Semiconductor Physics*. Moscow, Mir Publishers, 1978 (English translation).
8. KONYUSHKOV, G. V., ZOTOV, B. M., MERKIN, E. I. *Ferrites and Their Joining to Metals and Ceramics*. Energiya, Moscow, 1979 (in Russian).
9. KRASULIN, Yu. P. *Solid-Phase Interactions of Metals and Semiconductors*. Nauka, Moscow, 1971 (in Russian).
10. BOKIY, G. V. (ed.). *Crystal-Chemical, Physico-Chemical and Physical Properties of Semiconductors*. Standard Publishers, Moscow, 1973 (in Russian).
11. LYUBIMOV, M. L. *Glass-to-Metal Seals*. Energiya, Moscow, 1968 (in Russian).
12. MELNIKOV, P. S. *A Handbook of Electroplating in Machine-Building*. Mashinostroyeniye, Moscow, 1979 (in Russian).
13. METELKIN, I. I., PAVLOVA, M. A., POZDEYEVA, N. V. *Welding of Ceramics to Metals*. Metallurgiya, Moscow, 1977 (in Russian).
14. ROSSOSHINSKY, A. A. (ed.). *Pressure Microwelding*. Tekhnika, Kiev, 1971 (in Russian).
15. PAVLUSHKIN, N. M. (ed). *Glass. A Handbook*. Stroyizdat, Moscow, 1973 (in Russian).
16. *Making of Gaps in Ferrite and Magnetic Recording Heads by Vacuum Diffusion Bonding*. In: "Vacuum Diffusion Bonding of Metals, Alloys and Nonmetals", ed. by N. F. KAZAKOV, PNILDSV, Moscow, 1973, pp. 223-229 (in Russian).

16

Quality Control of Diffusion-Bonded Joints

Yu. A. Timoshenkov and *V. G. Lomonosov*

16.1 General

A quality control scheme as applied to welding has to do with various techniques, procedures, managerial and engineering measures, and appropriate physical facilities. Its objective is to assure the desired quality and reliability of welded joints and also to determine their fitness under service conditions. In setting up a particular quality control scheme, the governing considerations should be the requirements that a particular class of welded joints are to meet and also those stated in applicable standards. A detailed list of standards used for this purpose in the Soviet Union will be found in [18].

In the field of diffusion bonding, use is made both of standard techniques, procedures and facilities, and of those developed specifically for the diffusion process. This chapter will be concerned with matters of quality control related to the specific nature of diffusion-bonded joints. A more detailed description of standard testing procedures (both destructive and nondestructive) can be found in many sources some of which are listed in the bibliography at the end of this chapter [4, 6, 8, 11, 12, 17, 18, 21].

7. KIREEV, P. S. *Semiconductor Physics*. Moscow, Mir Publishers, 1978 (English translation).
8. KONYUSHKOV, G. V., ZOTOV, B. M., MERKIN, E. I. *Ferrites and Their Joining to Metals and Ceramics*. Energiya, Moscow, 1979 (in Russian).
9. KRASULIN, Yu. P. *Solid-Phase Interactions of Metals and Semiconductors*. Nauka, Moscow, 1971 (in Russian).
10. BOKIY, G. V. (ed.). *Crystal-Chemical, Physico-Chemical and Physical Properties of Semiconductors*. Standard Publishers, Moscow, 1973 (in Russian).
11. LYUBIMOV, M. L. *Glass-to-Metal Seals*. Energiya, Moscow, 1968 (in Russian).
12. MELNIKOV, P. S. *A Handbook of Electroplating in Machine-Building*. Mashinostroyeniye, Moscow, 1979 (in Russian).
13. METELKIN, I. I., PAVLOVA, M. A., POZDEYEVA, N. V. *Welding of Ceramics to Metals*. Metallurgiya, Moscow, 1977 (in Russian).
14. ROSSOSHINSKY, A. A. (ed.). *Pressure Microwelding*. Tekhnika, Kiev, 1971 (in Russian).
15. PAVLUSHKIN, N. M. (ed). *Glass. A Handbook*. Stroyizdat, Moscow, 1973 (in Russian).
16. *Making of Gaps in Ferrite and Magnetic Recording Heads by Vacuum Diffusion Bonding*. In: "Vacuum Diffusion Bonding of Metals, Alloys and Nonmetals", ed. by N. F. KAZAKOV, PNILDSV, Moscow, 1973, pp. 223-229 (in Russian).

16

Quality Control of Diffusion-Bonded Joints

Yu. A. Timoshenkov and *V. G. Lomonosov*

16.1 General

A quality control scheme as applied to welding has to do with various techniques, procedures, managerial and engineering measures, and appropriate physical facilities. Its objective is to assure the desired quality and reliability of welded joints and also to determine their fitness under service conditions. In setting up a particular quality control scheme, the governing considerations should be the requirements that a particular class of welded joints are to meet and also those stated in applicable standards. A detailed list of standards used for this purpose in the Soviet Union will be found in [18].

In the field of diffusion bonding, use is made both of standard techniques, procedures and facilities, and of those developed specifically for the diffusion process. This chapter will be concerned with matters of quality control related to the specific nature of diffusion-bonded joints. A more detailed description of standard testing procedures (both destructive and nondestructive) can be found in many sources some of which are listed in the bibliography at the end of this chapter [4, 6, 8, 11, 12, 17, 18, 21].

16.2 Defects in Diffusion-Bonded Joints and Their Causes

For lack of proper supervision during the preparatory steps, it may so happen that unwanted impurities (oxides, grease, other organic substances, and solid foreign bodies) are left on the mating surfaces. As a rule, their presence interferes with establishing a complete contact between the pieces to be joined. In consequence, the progress of bonding is impeded and various defects appear in the joint. Some defects may also arise from failure to follow the prescribed bonding procedure or to maintain the specified bonding conditions. The most typical defects in diffusion-bonded joints are incomplete bonding, cracks, and excessive bulk deformation. Other defects may also develop, and they will be discussed in this chapter as well.

Incomplete bonding manifests itself as unbonded spots from a few tenths of a micrometre to several micrometres wide (in the direction perpendicular to the joint surface) and from a few tenths of a square millimetre to several square millimetres in area. In some cases, unbonded spots may be comparable in size with individual grains. An unbonded spot may be filled by in-leaking air, an inert gas (if one is used for the shielding purposes), oxides or contaminants that have not had time to diffuse into the pieces being joined. As a rule, unbonded spots are narrow, scattered all over the joint area, and do not emerge to the external surface of the assembly. Sometimes, unbonded spots join to form strings which appear on a microsection as intermittent lines running along the joint surface. Cases have been reported in which unbonded spots formed networks of flaws each up to 10 μm long. Incomplete bonding may be caused by an unhappy choice of work design, improper placement in the bonding chamber, a poor design of the jig or fixtures used, substandard surface preparation for joining, departures from the prescribed bonding conditions (an insufficient bonding temperature, bonding pressure and/or holding time), and failure to maintain the specified composition of the atmosphere in the bonding chamber. For example, strings of unbonded spots will form in the diffusion-bonding of 35ХГСА steel to itself if the bonding temperature or bonding pressure is too low or the surfaces have been poorly prepared. An increase in holding time will usually heal up these defects.

In the diffusion bonding of metals and alloys, three basic types of structure may be formed in the bond zone. Type 1 displays two interfaces — one on each side of the joint, with the metal included between them differing in structure from that of the parent metal(s). Type 2 shows a contact line about as wide as a grain boundary. Type 3 displays no traces of interface. In each type of structure, defects will usually be different from those likely to appear in the other types. As an example, consider bonding a Ст.3 steel specimen to another Ст.3 steel specimen. In an experiment, a bonded joint with a type 1 structure showed an interface line up to 1 μm wide or comparable in width with a grain boundary, in addition to unbonded spots up to tens of micrometres wide. In a type 2 joint, there were recrystallized areas in the contact zone and unbonded spots 1-2 μm wide and up to 15 μm in diameter merged into a string or band. In a type 3 joint, the joined metals had diffused into each other over almost all of the contact area; in the remainder an interface line could be seen,

almost identical with intergranular boundaries, and unbonded spots 2 μm wide and 6 μm in diameter. Metallographic analysis showed that the unbonded spots were obviously smaller than grains, and there were a large number of microscopic unbonded spots.

Unbonded spots with an area of over 0.5 mm^2 and more than 1 μm wide can substantially impair the strength and fatigue life of bonded joints. Apart from their area and width, as studies have shown, the number of unbonded spots can also affect joint strength. In one case involving 20ХГСА steel specimens, when the bonding pressure was reduced to one-fifth of its original value, the ultimate tensile strength of the assembly decreased to one-ninth, and this was traced to the increase in the size and number of unbonded spots. In another case, the number of unbonded spots in steel-45 specimens was increased 2.5 times, and the ultimate tensile strength dropped to nearly half its original value. Unbonded spots are objectionable also because they may serve as nuclei of cracks developing under load.

Cracks, especially those running to the surface, are extremely dangerous. They may develop from macro- and microcracks with the result that the interior of the bond is open to ingress of contaminants, stresses may rise to their critical values, and the cracks will further propagate. Cracking is usually provoked by an excessive rate of heating and cooling, an excessive bonding pressure and/or temperature, and an improper choice of design or configuration for the workpieces so that appreciable internal stresses may arise. Still another cause of cracking may be the sharp difference in thermal expansion between the materials being joined.

An excessive bulk deformation occurs when the bonding pressure is chosen such that the yield point of the joined materials is exceeded.

In the diffusion bonding of titanium and titanium alloys, failure to maintain the optimal bonding conditions may lead to defects in the form of pores and the remnants of an oxide or grease film, which are oriented along the original interface. Defects in the form of oxide films (several micrometres thick) have also been observed in joining 12Х18Н10Т steel to itself and other grades. Oxide films can appear at the interface because the mating surfaces are poorly prepared for bonding. For this reason, a microsection of a Mo-Mo composite diffusion-bonded at a temperature of 1 373 K, a pressure of 15 MPa, a holding time of 20 min, and a vacuum of 6.5×10^{-2} Pa displayed dark oxide films. In some cases, it is not mandatory to remove oxide films from the mating surfaces in order to make a quality joint. This is true, for example, of glass-to-metal seals and metal-to-ceramic seals, where the presence of a thin oxide film favours the bonding process.

Sometimes, diffusion-bonded joints display a defect which may be called a false bond. It occurs if the joined metals have not diffused into each other for a sufficient depth, which happens when the prescribed bonding conditions have not been maintained. In one case involving a stack of copper plates (each plate being 0.2 mm thick and the entire stack 12 mm high), a false bond instead of a true one occurred because the bonding temperature was too low. From a microsection it was observed that each plate was separated from its neighbour by an oxide coating 2 μm thick. In the case of a false bond, the plates of a composite can be readily detached from one another. In contrast, when a true bond is formed, it takes an appreciable effort equal in magnitude to the strength of the materials in order to break the joint.

Marked changes in the structure of the bond zone in comparison with the parent materials may also be classed as defects. In making titanium-titanium composites grain growth is frequently observed. Sweating (surface melting) can sometimes occur if the pieces are heated to a temperature close to their melting point(s). This may be caused by a poor design of the heat source or when it is incorrectly positioned relative to the workpieces (for example, the inductor turns may be placed too close to the work). Misalignment, a further form of defect, can be caused by an inadequately designed jig or fixture and the improper placement of the pieces in it.

As follows from the foregoing, one and the same cause can produce several defects differing in nature. Obviously, more than one defect can develop in a specimen at the same time, impairing the quality of the joint and of the assembly by a sizeable amount. In the general case, the presence of a small number of defects is not a sufficient reason for rejecting a product, especially if it meets relevant service requirements. However, the detection of even minute flaws serves the purpose of improving the bonding techniques and procedures.

16.3 Objectives and Scope of Quality Control in Diffusion Bonding

A quality control scheme must be capable of the timely detection of all defects and of their causes so that the likely departures from the prescribed bonding conditions can be corrected. To this end, quality control must be effected at every step in

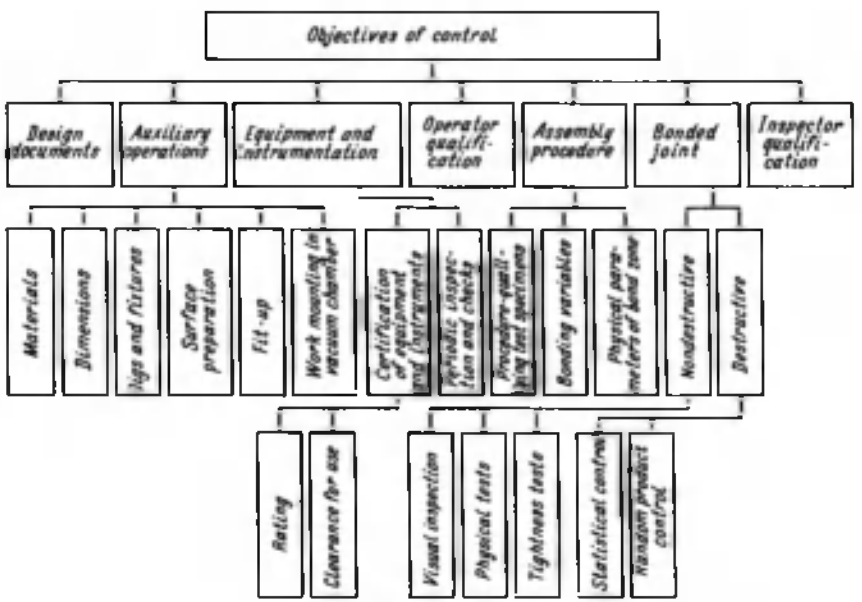

FIG. 16-1 Quality assurance program for diffusion bonding

the operation — from receipt of materials for bonding throughout all processing to the final item.

The scope of quality control in diffusion bonding usually varies with the end purpose to be served by a weldment, how critical it is, and the manner in which the operations involved in diffusion bonding are organized. In diagrammatic form, it is illustrated in Fig. 16-1. The activities shown are intended to create conditions under which the risk of defects is minimized and the quality of the finished product is assured. The scheme is applicable to critical products, so some activities can be skipped if the product on hand is to meet less stringent service requirements. For example, in the case of small-size turbines the principal requirements to be met by joints are strength, freedom from cracks, and absence of incomplete bonding; this can be ascertained by mechanical, ultrasonic, and fluorescent testing. Since vacuum tightness is not important in this particular case, the associated test should be omitted from the program. In the case of heat exchangers, vacuum tightness is a vital parameter, so this form of testing is fundamental.

16.4 Control of Factors and Operations Preceding Diffusion Bonding

So that all-over nondestructive testing can be applied to diffusion-bonded items, the product designer must make them readily manageable to quality control. To this end, all drawings and specifications must be checked from a testing point of view already at the design stage, with the participation of experts in diffusion bonding and quality control. The quality and composition of incoming materials should be checked against specifications as prescribed by relevant regulations. The point is that the bonding conditions optimal for one chemical analysis may prove completely unsuitable for another. For example, one set of bonding variables may be good for joining AJ1 9 cast aluminium alloy to BT1-0 titanium alloy, but it will most certainly be unsuitable to join another aluminium alloy, AJ1 26, to the same titanium alloy. In the examples cited, this will occur because the two aluminium alloys differ in the silicon content.

Geometry testing is important because the departure from some parameter may lead to defects that constitute a cause for rejection. For example, if pieces from brittle materials (ceramics, glass,etc.) are not polished flat to within the specified limits, the applied pressure will be distributed over the contact area nonuniformly and cause cracking in the joint. In the case of plastic materials, the same factor will usually produce an excessive bulk deformation. Failure to keep surface roughness within specified limits may be responsible for a poor actual contact between the mating surfaces. Therefore, the degree of roughness must be specified well in advance, so that a quality bonded joint can be made. For many metals and their alloys, it will suffice to machine the mating surfaces to a roughness of 2.5 μm. Greater roughness will usually impair joint strength appreciably.

The dimensions, configuration and surface geometry of the pieces coming in for diffusion bonding can be checked with a wide range of gauges, micrometers, calipers, tool-room microscopes, surface roughness indicators, surface roughness recorders, etc.

Prior to bonding, it is important to check the devices used to transfer bonding pressure to the pieces and to hold them in the correct position. Above all, it is essential to check the material(s) of the devices. If the bonding temperature does not exceed 1 050 K, the devices can be fabricated from a corrosion-resistant steel (such as 12X18H10T), whereas at a higher bonding temperature they should be made of molybdenum or some other refractory metal or alloy. The next step is to check the dimensions of the devices and to see if their contacting surfaces are given a coat of parting substance which will prevent freezing to the work. If the parting coat has come off, the device should be baked in an atmosphere of, say, wet hydrogen at 1200-1250 K for 2-3 h. Another item to be checked prior to bonding is provision of an opening or a recess in the device for a thermocouple. During fit-up, it is necessary to make sure that the contacting surfaces of the device conform to the contour of the workpieces, that the pieces are properly aligned relative to each other, that a pressure equalizer (a piece of plastic material) is inserted, and that proper clearances are left between the elements provided to limit the distortion of the workpieces. In the case of complicated shapes, they can be tested for an even bearing all around by smearing them with suitable paints.

The reliable and consistent operation of the various elements in a diffusion-bonding machine is a vital aspect of the entire process. Therefore, the bonding facilities to be used should be checked and tested so that the specified bonding variables can be reproduced with the desired repeatability. This includes periodic inspection of all the systems (the heat source, the bonding pressure system, the vacuum system, the cooling system, etc.) of the machine, scheduled maintenance, and re-certification in the course of which the machine and its systems are tested for performance against applicable specifications. The instruments (vacuum gauges, dynamometers, potentiometers, ammeters, etc.) mounted on a diffusion-bonding machine are subject to periodic calibration and, if necessary, repair.

The proficiency of bonding-machine operators and quality inspectors may directly affect the quality of bonded joints, so their training and skill level should meet a number of specific requirements [18].

16.5 Monitoring Bonding-Process Variables

All the process variables of diffusion bonding (bonding temperature, bonding pressure, holding time, vacuum) are rigorously specified in magnitude and duration. The manner in which they may vary in the course of bonding depends on the work design, the materials used, and the requirements to be met by the product. The manner in which the bonding variables change in the course of diffusion bonding is illustrated in Fig. 16-2. As is seen, the operation should be commenced at the steady-state values of the variables (stage *A-B*) — it is in this stage that the events affecting the quality take place. It is highly advisable to record all the bonding variables during the operation, especially when trying out or checking the procedure. The performance data of some of the instruments used in the Soviet Union to monitor and record the bonding variables are listed in Table 16-1.

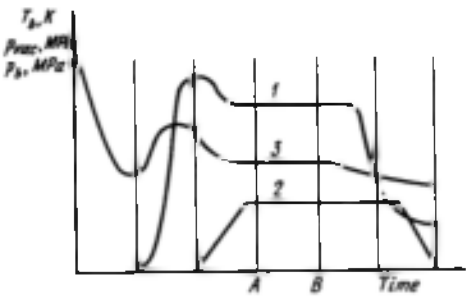

FIG. 16-2 Time variations in diffusion bonding variables
1 — bonding temperature, T_b; *2* — bonding pressure, p_b; *3* — vacuum in chamber, p_{vac}

TABLE 16-1

Bonding variable	Instrument	Range
Bonding temperature	Thermocouples [16]:	
	Chromel-Alumel	100-1600 K
	Chromel-Copel	100-900 K
	Tungsten-Rhenium	300-2800 K
	Pyrometers [15]:	
	Disappearing-filament	1100-6300 K
	Same, of higher accuracy	1000-6300 K
	Same, reference	1200-6300 K
	Photoelectric	800-4300 K
	Colour	1700-3100 K
	Radiation	700-3800 K
Bonding pressure	Dial dynamometers	2×10^2-2×10^3 N 1×10^3-1×10^4 N 1.8×10^3-3×10^4 N 5×10^3-5×10^4 N
Vacuum in bonding chamber	Ionization-thermocouple vacuum gauges [2]	6.5×10^{-6}-2.5×10^{1} Pa 6.5×10^{-6}-1.3×10^{2} Pa
Holding time (duration of diffusion treatment)	Timer, clock	

16.6 Measurement and Control of Physical Variables in the Bond Zone

In recent years, the prevailing trend has been towards active control of the variables in diffusion bonding. To this end, the bonding variables are measured and adjusted (if necessary) in relation to some physical variables. The relationships to be maintained are established in advance. Figure 16-3 shows the set-up developed at the PNILDSV for the purpose (USSR Inventor's Certificate No. 454103). The reference physical quantity is the rate of leakage continuously monitored in the course of bonding by measuring the flow of helium across the bond zone. The pieces to be joined, *3*, are mounted in a vacuum chamber (consisting of members *1* and *2*), and are sealed off with gaskets *4*, except for the bond zone. The chamber is exhausted to a vacuum of 10^{-1}-10^{-3} Pa, the workpieces are heated by an inductor, *6*, to a temperature equal to 50-80% of the fusion point of the lowest-melting-point metal, and a bonding pressure of up to 10^8 Pa is applied. The inner space formed by the joined pieces and the bottom of the chamber, *2*, is placed in communication with a helium source through an opening in the chamber bottom. If the bond is not tight, some helium will find its way out of the inner space into the bonding chamber and, hence, into a mass spectrometer, *5*, which records the rate of leakage.

Once a leak is detected, adjustments are applied to the bonding variables, namely, the bonding temperature, bonding pressure and holding time are increased separately or together until the leak is stopped. This method has successfully been used to monitor the diffusion bonding of 22XC ceramic to titanium and copper (the products were hollow pieces).

A further step in this direction is the technique developed to detect inner flaws in diffusion-bonded joints (USSR Inventor's Certificate No. 565793). Now, the inner space of the assembly is connected by a line to a cylinder holding a mixture of helium and hydrogen, and the bonding chamber is connected to a gas analyzer. After the chamber is exhausted, a cold-pressing load is applied, and the assembly is

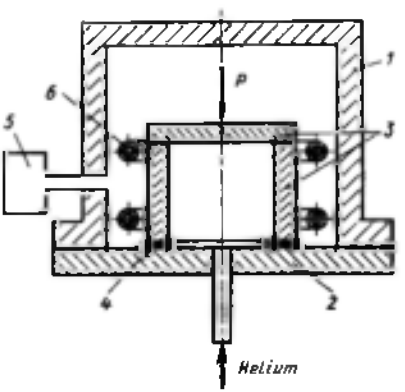

FIG. 16-3 Setup to monitor the diffusion-bonding variables

heated. Then the final bonding pressure is applied and the gas mixture is admitted to the inner space. The gas analyzer records the flow rate of gas mixture across the bond zone. When the helium flow ceases, this is an indication that an actual contact has formed. When the hydrogen flow attains a steady-state value, this is an indication that the micropores in the joint have been healed by diffusion. Now, the bond is practically equal in properties (say, vacuum tightness) to the parent materials. It is an easy matter to determine by experiment the ultimate strength, the degree of deformation, and the bonding variables necessary to obtain such a joint. This technique has been used to test the quality of several composites such as copper-copper, steel-steel, and metal-ceramic.

In practice, it is more convenient to monitor the process of diffusion bonding by measuring the deformation of the components being joined [7]. The bonding operation is discontinued when a predetermined deformation has been attained. A functional relationship is established in advance between the deformation and the quantities related to the service performance of the bonded joint (strength, impact toughness, etc.). This technique has been continuously applied to testing a wide range of bonded joints, such as nickel-nickel, titanium alloy-titanium alloy, and aluminium alloy-titanium alloy.

As an alternative, the reference quantity may be the electric resistance of the bond zone. With this technique, a current is passed through the bond zone, and a voltage drop is measured. The voltage drop across the material adjacent to the joint is higher than across the parent metal, because its resistance is raised by the presence of defects such as unbonded spots, oxide inclusions, etc. The value of resistance depends on the shape, size and number density of defects [10, 20]. The relationships between resistance, strength and other service performance indicators are correlated with holding time (duration of diffusion treatment) (Fig. 16-4). To avoid errors in measuring the resistance due to the instability of contact between the probe and the work, it is usual to employ a four-terminal test setup. The connecting leads are usually made of the same material as the workpieces so as to minimize the effect of the thermo-emf generated between the work and the leads in the high-temperature zone. The resistance can be measured with a microohmmeter or a potentiometer. This technique has successfully been applied to several metal systems, such as steel-steel, steel-copper + nickel, steel-copper, steel-nickel, etc.

In the case of bimetal composites, the purpose of control can be served by the thermocouple formed by the components themselves. The time dependence of the thermo-emf generated at the junction is measured and compared with a reference (USSR Inventor's Certificate No. 688873).

In the diffusion bonding of dielectric materials (glass, ceramics, quartz, etc.) with a metal interlayer for which the recrystallization temperature is higher than the bonding temperature, resort can be made to the technique illustrated in Fig. 16-5 (USSR Inventor's Certificate No. 585936). The pieces to be joined, *1*, are fitted up with a current-conducting interlayer *2* (which may be nickel foil) with an area exceeding that of the workpieces, and the assembly is placed in a vacuum chamber, *3*. On heating and compression, an interface is formed between the deformed and nondeformed parts of the interlayer, with the nondeformed part lying outside the

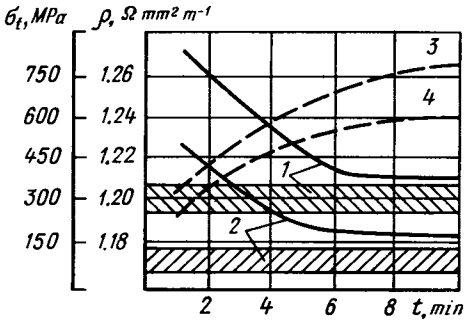

FIG. 16-4 Time variations in resistance (*1*, *2*) and strength (*3*, *4*) in the diffusion bonding of steel-45 (*1*, *3*) and Ст.3 steel (*2*, *4*). Shaded areas give the resistance of the parent metal

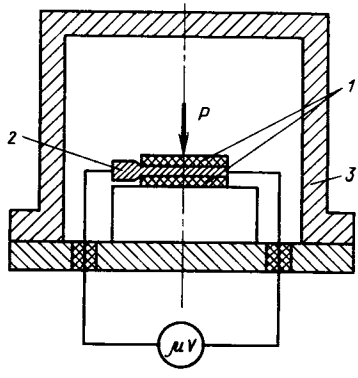

FIG. 16-5 Setup to monitor the diffusion bonding of dielectric materials with a current-conducting interlayer

bond zone. As a result, a highly sensitive source of thermo-emf is formed, which can be measured by a microvoltmeter connected to the deformed and undeformed parts of the interlayer. This emf is a function of the bonding pressure applied. This technique is advantageous over those based on measuring the bonding pressure with a pressure gauge or a dynamometer. The latter instruments are rather inaccurate, so they measure the bonding pressure with an error, and this leads to spoilage in diffusion bonding. With pressure gauges, the source of error lies in the fact that they do not respond to the friction existing throughout the power drive. With dynamometers, the error is caused by seizure in the rubbing parts of the dial indicator. The technique based on thermo-emf yields the true pressure at the mating surfaces.

In one practical case, cylindrical silicon pieces 10 mm in diameter and 1 mm high were joined by diffusion bonding with a nickel foil 0.2 mm thick. The bonding conditions were $T_b = 500$ K, $p_b = 23$ MPa, $t_b = 20$ min, and $p_{vac} = 6 \times 10^{-2}$ Pa. The interlayer projected for a length of 10 mm. The measurement of the bonding pressure with a microvoltmeter proved four times more accurate than with a pressure gauge.

The duration of the bonding operation can be set by determining the deformation of the material from the electromagnetic radiation emitted at the bond zone when the workpieces are compressed (USSR Inventor's Certificate 504140). The energy, power or other parameter of the radiation can be measured with a radiation detector placed inside the bonding chamber. The relation between the deformation required to make a quality joint and the measured radiation variable is established in advance. During the bonding operation, watch is kept on the readings of the radiation indicator, and the bonding operation is discontinued when the meter reads the predetermined value of the chosen variable.

16.7 The Use of Test Specimens

An important step in assuring the quality of diffusion-bonded products is periodic tests on procedure-qualifying test specimens. These specimens are easy to make and can readily be tested and measured. Given the same conditions (the same quality of materials, the same quality of surface preparation, the same bonding variables, etc.), the data obtained by measuring test specimens may rightfully be extended to actual weldments. The quality of bonded joints in test specimens is evaluated on the basis of the tests and measurements conducted as required for welded products. Mechanical strength is not the only criterion of joint quality. Sometimes it may be essential that one of the metals being joined should retain its electrical properties at the joint or that diffusion-bonded quartz elements should preserve their optical properties and geometry at the bond, etc. In some cases, diffusion-bonded products are not required to have a high mechanical strength at all. As an example, in the case of components for the electrodes of electrolysis cells fabricated from porous or gauze materials by diffusion bonding the most important requirement is the electrochemical characteristic at various current densities.

Once the above data have been collected, they should be analysed by methods of mathematical statistics. The objective of the analysis is to form the sample deviation and the sample mean square, for which purpose steps are taken to determine the size of the sample (the number of test specimens or measurements to be made) so as to assure an appropriate accuracy. This is a standard procedure examined in detail in many publications [8].

It is to be noted that even if test specimens and actual products are diffusion-bonded under identical conditions, they may still differ in joint quality for several reasons. Among other things, in the case of products substantially larger in size than the test specimens used, the heat input can be nonuniformly distributed over the mating surfaces or the bonding pressure can be nonuniformly transferred. Generally, test specimens and actual products differ in their contour and shape, so the bond cannot be formed under truly identical conditions.

In some cases, especially where critical bonded products are involved, the destructive testing of specimens and products should be preceded by their nondestructive testing and some other procedures in order to correlate the variables of interest.

In the Soviet Union, basic techniques and procedures for determining the mechanical properties of welded joints and joint elements are defined by USSR State Standard GOST 6996-66. Standards have been established for tests in tension, impact toughness, corrosion resistance, etc. [18].

16.8 Quality Control of Bonded Products

In order to assure product quality with any of the tests or control forms defined above, it is important to choose them discriminately so as to suit the design and product on hand (especially in the case of active control forms). The factors affecting the choice of a particular form of test or control are the product design, the product materials, the service requirements for joints and products, the defects likely to oc-

cur in the joint, and the merits and demerits in comparison with the rival techniques and procedures.

Weld testing by X- and gamma-rays widely practised in fusion welding is inapplicable to diffusion bonding because of the small size of the defects that usually occur. The sensitivity of inspection with X- and gamma-rays is 0.5-2%, whereas for the defects occurring in diffusion bonding it should be 0.01-0.0001%. Such defects can be detected with ultrasound. Ultrasonic testing is equally applicable to weldments of both simple and complicated shape, including bimetal assemblies fabricated by diffusion bonding (Fig. 16-6). It cannot, however, be used for small parts or components with a roughness of more than 2.5 μm. Also, the capability of ultrasonic flaw detection is limited by the frequency range of existing ultrasonic testers.

From the viewpoint of detectability by ultrasonic inspection, the defects in diffusion-bonded joints may arbitrarily be classed into three types, namely macrodefects each with a surface area of over 1 mm^2 and more than 10 μm wide; microdefects each with a surface area of 0.5-1 mm^2 and more than 1 μm wide; and microdefects each with a surface area of less than 0.5 mm^2 and less than 1 μm wide (usually located within grains due to the migration of grain boundaries) [5]. At present, ultrasonic testing can reliably detect flaws of the first two types which are most objectionable from the service point of view. They can be detected with several models of commercially available instruments, such as the Soviet-made DUK-66 with an operating frequency of up to 10 MHz, the West-German USIP-11, the American Nanoscope-412, and the Polish Unipan-510 operating at far higher frequencies.

An example of using ultrasonic inspection for flaw detection is the bronze-steel assembly (a bronze bushing in a steel cylinder) shown in Fig. 16-6. Flaws are detected by generating Lamb waves in the metal by a transducer assembled of short piezoelectric plates, with reflections received by the same or a second similar transducer. The electronic part of the instrument is built around a standard flaw detector. The smallest unbonded spot that can be detected with the instrument is 3 mm^2. Cracks in the bronze and in the transition layer can also be detected reliably [19].

The procedure of flaw detection is as follows. To begin with, the test instrument is fitted with a transducer whose size is comparable with the diameter of the product

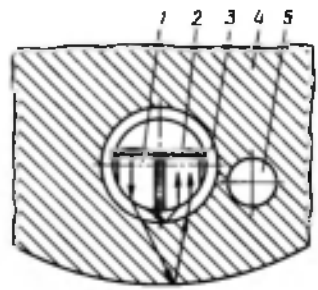

FIG. 16-6 Setup for the ultrasonic testing of a bronze-steel bonded joint
1 — transmitting ultrasonic transducer; *2* — receiving ultrasonic transducer; *3* — bronze bushing; *4* — steel cylinder; *5* — fabrication hole giving rise to stray reflections

to be tested, and adjusted by reference to a specimen having a standard bore and a flat bottom. The sensitivity is set at a level sufficient for an automatic flaw indicator to operate. Then the transducer is introduced into the bushing, and its surface is scanned in a helical pattern. The defects thus detected are presented on a cathode-ray tube, and the automatic flaw indicator lights up if the defects exceed the sensitivity level. In the case of stray reflections (such as from the fabrication hole, *5*, in Fig. 16-6), the results can be double-checked by measuring the electric resistance of the bond zone by the method described earlier.

The resistance method is sensitive towards defects in the form of discontinuities lying at a depth of several millimetres, the sensitivity being a maximum when the terminals and the probes are placed closest to the region containing a defect. The procedure for measuring the electric resistance of the bond zone is as follows. A reference specimen is first prepared in which the bonded joint meets all relevant requirements, and the resistance of the test joint is measured. Then the resistance of the actual joint is measured, and the two results are compared. In joints between identical metals, the resistance of the bond zone ought not to exceed that of the parent metal. It has been found that the resistance of a metal can be changed by degassing and owing to the re-distribution of impurities on heating in the vacuum chamber.

TABLE 16-2

Method	Minimum depth of crack, μm	Minimum width of crack, μm	Application
Ultrasonic	100	1	Magnetic and nonmagnetic materials (except small odd-shaped and rough-surfaced parts); internal, subsurface and external defects
Resistance	200	10	Current-conducting materials; defects in the form of discontinuities lying at a depth of up to 4 mm
Eddy-current	100	1	Magnetic and nonmagnetic materials (except small odd-shaped parts); subsurface and external defects
Magnetic-particle	10	1	Magnetic materials; external and subsurface defects
Fluorescent	40	20	Magnetic and nonmagnetic materials; surface defects
Fluorescent magnetic-particle	5	0.1	Magnetic materials; external and subsurface defects
Dye-penetrant	20	1	Magnetic and nonmagnetic materials; surface defects
Visual	—	100	Same

The liquid-penetrant, eddy-current and magnetic-particle methods of nondestructive testing widely used to detect external and subsurface flaws in magnetic, nonmagnetic and other materials are equally successful in the testing of diffusion-bonded products. Data on the sensitivity and uses of the above methods of flaw detection are listed in Table 16-2 [4].

As already noted, the use of any one method may prove insufficient for various reasons. It is therefore advisable to employ a combination of several methods. In the example illustrated in Fig. 16-6, the ultrasonic method was supplemented with the resistance method.

In one practical case, the ultrasonic testing of titanium plates diffusion-bonded to an aluminium plunger failed to identify the nature of the defects: unbonded spots could not be told from cracks. In the aluminium, the cracks produced by heating and cooling caused the automatic flaw indicator to signal an unbonded spot.

In another case involving a joint between titanium and an aluminium alloy, the identification of unbonded spots called for an additional metallographic examination of the defective areas brought out by ultrasonic testing.

Frequently, products which contain diffusion-bonded joints are subjected to tests for leaks, vacuum tightness, corrosion resistance, and thermal cycling. This is done by the usual techniques and procedures.

Most often, internal stresses in various products, including welded ones, are determined in practice by a mechanical method (which calls for removal of layers electrochemically) and by X-raying. Both are described in detail in [1].

A more recent addition is the deformation characteristic (or electronic) method (USSR Inventor's Certificate No. 327379). This is a nondestructive testing technique utilizing the nonlinear behaviour of a solid within the elastic region in the presence of internal stresses. The use of the method calls for knowledge of the temperature dependence of the natural frequency of vibrations of the weldment (test specimen) (including longitudinal, bending and twisting vibration). This dependence is called the deformation characteristic (Fig. 16-7). Frequency has been chosen for reference because it can be measured in practice to a very high degree of accuracy. At the equilibrium temperature, T_{eq}, the slope of the deformation characteristic (the tangent of the angle made by a straight line drawn touching to the curve) is equal to the temperature coefficient of frequency in the absence of internal

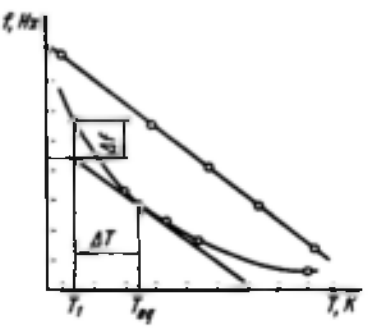

FIG. 16-7 Deformation characteristic used to determine internal stresses in a bonded joint

stresses. This coefficient can readily be looked up in reference sources or determined by experiment after the specimen is heat-treated to make its behaviour linear (in which case, the deformation curve will be linear).

The procedure is as follows. The specimen is clamped in a dial-type vibration-inducing device in a manner that avoids plastic deformation which might lead to additional internal stresses. The assembly is set up on a cast-iron or steel plate and loaded into a constant-temperature cabinet. After the temperature is the same throughout the bulk of the specimen, its natural frequency of vibration is measured. The temperature is then raised by an increment, the specimen is held until the temperature has equalized, and the frequency is measured again. This sequence is repeated several times, and the measurements are plotted. In order to determine the equilibrium condition a tangent is then drawn (according to the temperature coefficient of frequency). Using the characteristic, the magnitude of internal stresses is found by the following equation

$$\sigma = k(E/\mu)(\Delta f/f_1)$$

where k is the coefficient dependent on the mode of vibration used and the manner in which the specimen is clamped; E is Young's modulus; μ is Poisson's ratio; f_1 is the natural frequency at a given temperature; Δf is the nonregular component of frequency increment relative to the equilibrium condition.

The deformation characteristic method has been successfully used to monitor internal stresses in designing techiques and procedures for the diffusion bonding of quartz to quartz, copper to molybdenum, etc.

In diffusion-bonded ceramic-metal composites, the residual stresses can readily be measured with strain gauges. In an experiment, strain gauges were cemented to the surface of a ceramic cylinder bonded to a copper disc. All gauges were laid out near the bond zone all the way round the perimeter of the cylinder and connected in series into the half-bridge of an electronic meter. The instrument was then balanced by reference to one of the gauges. Measurements were made after each composite had been cut with a diamond wheel through the ceramic at the joint, so that all internal stresses were relieved. The difference between gauge readings before and after cutting gave a measure of the elastic deformation of the ceramic. The circumferential and the most dangerous bending stresses acting in the bond zone were then found by calculation.

In optically transparent materials, residual stresses can effectively be determined by the polarization or photoelasticity method. It has been applied to testing metal-to-glass seals, quartz-to-quartz joints, etc. [9]. Several types of polariscopes-polarimeters can be used for the purpose.

ADVANCED METHODS OF WELD ANALYSIS. Recently, the Central Research Institute for Heavy Machine-Building (USSR) has developed several techniques for weld inspection based on ultrasonics in combination with pattern recognition by a computer. It will undoubtedly prove effective because, as Academician Nikolayev has shown, the performance of weldments depends above all on the shape (pattern) of defects. Another advanced technique for studies in the field of diffusion bonding and inspection of finished weldments is one utilizing the emission of elastic waves,

known as the acoustic emission method. Diffusion bonding is accompanied by a number of dynamic phenomena (such as plastic deformation, rupture of internal bonds, etc.) which cause the emission of elastic waves, and these can be registered by acoustical means. An advantage of this form of flaw detection is that a defect acts as a signal source in the course of the bonding operation [3]. The acoustic emission method has been used to monitor the formation of diffusion bonds and to evaluate the quality of weldments. In the diffusion bonding of copper to beryllium, for example, it has been found that the dependence of acoustic-emission signals on heating and cooling times can serve as a guide with which to follow the development of relaxation process in the bond zone, and the formation and dispersal of intermetallic layers [14]. Holographic flaw detection also holds out promise for its use in the quality control of diffusion-bonded joints. The experiments made to date have yielded positive results in the inspection of thin-walled assemblies [13].

Bibliography

1. BIRGER, I. A. *Residual Stresses*. Mashguiz, Moscow, 1963 (in Russian).
2. MENSHIKOV, M. I. (ed). *Vacuum Techniques. A Handbook*. Reklama, Moscow, 1975 (in Russian).
3. GRESHNIKOV, V. A., DROBOT, Yu. B. *Acoustic Emission*. Standard Publishers, Moscow, 1976, pp. 201-228 (in Russian).
4. DENEL, A. K. *Flaw Detection in Metals*. Metallurgiya, Moscow, 1972, p. 13 (in Russian).
5. AZAROV, N. T., MATYUSHKIN, B. A., LEVTOVA, K. A. *et al. A Study into the Potentialities of Ultrasonic Inspection of Titanium Alloy Joints Made by Solid-State Welding*. "Svarochnoye proizvodstvo" , 1979, No. 9, pp. 30-32 (in Russian).
6. KAZAKOV, N. F. *Diffusion Bonding of Materials*. Mashinostroyeniye, Moscow, 1976 (in Russian).
7. KARAKOZOV, E. S., LAVROV, B. A., BAGIN, V. M. *Active Control of Weld Quality and a Method for Determining Optimal Conditions for Hot Pressure Welding*. "Avtomaticheskaya svarka", 1978, No. 4, pp. 36-39 (in Russian).
8. VOLCHENKO, V. N., GURVICH, A. K., MAYOROV, A. N. *et al. Quality Control in Welding*. Mashinostroyeniye, Moscow, 1975 (in Russian).
9. KONYUSHKOV, G. V., KOPYLOV, Yu. N. *Diffusion Bonding in Electronics*. Energiya, Moscow, 1974, pp. 117-127 (in Russian).
10. KOTELNIKOV, A. A., BASHURIN, A. V., ZAICHENKO, N. P. *Quality Control in the Course of Diffusion Bonding*. "Avtomaticheskaya svarka", 1975, No. 11, pp. 34-35 (in Russian).
11. RUMYANTSEV, S. V., DOBROMYSLOV, V. A., BORISOV, O. I. *et al. Nondestructive Testing of Welded Joints*. Mashinostroyeniye, Moscow, 1976 (in Russian).
12. SAMOILOVICH, G. S. (ed.). *Nondestructive Testing of Metals and Products. A Handbook*. Mashinostroyeniye, Moscow, 1976 (in Russian).
13. LIPKIN, A. S., KARAKOZOV, E. S., VIGDORCHIK, S. A. *et al. Nondestructive Testing of Honeycomb Panels by Holographic Interferometry*. "Avtomaticheskaya svarka", 1976, No. 6, pp. 76-77 (in Russian).
14. PARAYEV, S. A., KARAKOZOV, E. S., LEBEDEV, N. V. *Testing of Diffusion-Bonded Joints in Copper-Beryllium Composites by the Acoustic Emission Method*. "Svarochnoye proizvodstvo", 1979, No. 9, pp. 29-30 (in Russian).

15. PREOBRAZHENSKY, V. P. *Measurements and Instrumentation in Heat Engineering.* Mir Publishers, Moscow, 1980 (English translation).
16. BYCHKOVSKY, R. V. (ed.). *Instruments for Temperature Measurements by the Contact Method.* Higher School, Lvov, 1979 (in Russian).
17. KLYUEV, V. V. (ed.). *Instruments for Nondestructive Testing of Materials and Products. A Handbook.* Mashinostroyeniye, Moscow, 1978 (in Russian).
18. ZORIN, Yu. N. (ed.). *Welding in Mechanical Engineering. A Handbook.* Mashinostroyeniye, Moscow, 1979 (In Russian).
19. BOBROV, V. T., DUSHIN, V. A., ZVEREV, I. N. *et al. Ultrasonic Flaw Detection in Diffusion-Bonded Bimetal Composites of Complex Configuration.* "Tekhnologiya legkikh splavov", 1975, No. 1, pp. 54-60 (in Russian).
20. USHAKOVA, S. Ye., VOLKOV, Ye. S., KOVRIGUIN, V. I. *Quality Control of Vacuum Diffusion-Bonded Joints.* "Svarochnoye proizvodstvo", 1967, No. 10, pp. 29-31 (in Russian).
21. KHIMCHENKO, N. V., BOBROV, V. A. *Nondestructive Testing in Chemical and Petroleum Machine Building.* Mashinostroyeniye, Moscow, 1978 (in Russian).

17

Accident Prevention. Cleanliness in Vacuum Diffusion Bonding

N. F. Kazakov

17.1 General

In the Soviet Union, all operations in diffusion bonding and the training and briefing of personnel are subject to the Regulations for the Operation of Vacuum Diffusion Bonding Equipment. Clearance to operate diffusion bonding machines may only be given to persons no less than 18 years old, who have passed a medical check-up, been trained in the use of plant utilizing h.f. currents, certified by a qualification panel, issued a permit to operate electric equipment, and duly briefed in accident prevention.

Vacuum diffusion bonding as performed on machines using h.f. heat sources involves elevated temperatures, high voltages, electromagnetic radiation, and the use of liquefied gases. Therefore, attending personnel is open to the risk of burns, an electric shock, fire hazards, functional disorders in the nervous and cardio-vascular systems, and cloudiness of the crystalline lens of the eyes.

A separate area should be allotted to a diffusion bonding machine which should be installed in a sufficiently large room. No flammable liquids and materials (petrol, alcohol, acetone, paper, etc.) may be stored or used within the diffusion-bonding area. Dielectric mats should be laid on the floor at and around the diffusion bon-

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16. BYCHKOVSKY, R. V. (ed.). *Instruments for Temperature Measurements by the Contact Method.* Higher School, Lvov, 1979 (in Russian).
17. KLYUEV, V. V. (ed.). *Instruments for Nondestructive Testing of Materials and Products. A Handbook.* Mashinostroyeniye, Moscow, 1978 (in Russian).
18. ZORIN, Yu. N. (ed.). *Welding in Mechanical Engineering. A Handbook.* Mashinostroyeniye, Moscow, 1979 (In Russian).
19. BOBROV, V. T., DUSHIN, V. A., ZVEREV, I. N. *et al. Ultrasonic Flaw Detection in Diffusion-Bonded Bimetal Composites of Complex Configuration.* "Tekhnologiya legkikh splavov", 1975, No. 1, pp. 54-60 (in Russian).
20. USHAKOVA, S. Ye., VOLKOV, Ye. S., KOVRIGUIN, V. I. *Quality Control of Vacuum Diffusion-Bonded Joints.* "Svarochnoye proizvodstvo", 1967, No. 10, pp. 29-31 (in Russian).
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A separate area should be allotted to a diffusion bonding machine which should be installed in a sufficiently large room. No flammable liquids and materials (petrol, alcohol, acetone, paper, etc.) may be stored or used within the diffusion-bonding area. Dielectric mats should be laid on the floor at and around the diffusion bon-

ding machine. Connection to and disconnection from the power line and its supervision in service must be effected by duly qualified electricians.

It is important to check the electromagnetic field intensity each time there has been a change in the layout of supply cables, equipment, wiring or shielding, and also after repair. The diffusion-bonding area should be equipped with a plenum-exhaust ventilation system which, in special cases, must be supplemented with an air-conditioning system to maintain the desired microclimatic parameters. Foul air should be exhausted at the top of the room, and fresh air admitted to the working area.

Bonded parts and jigs, usually heated to 673 K, must be removed from the bonding chamber with tongs.

Parts to be joined should be degreased before they are delivered to the diffusion-bonding area. The room used for degreasing with organic solvents (acetone, ethyl alcohol and the like) should be equipped with a plenum-exhaust ventilation system as prescribed for industrial premises. The plenum-exhaust system must be capable of replacing foul air with fresh air at least 15 times an hour. The atmosphere in the diffusion-bonding area must periodically be tested for the impurity content to a schedule coordinated with the accident prevention department. Under relevant regulations in force in the Soviet Union, the acetone concentration in the area should not exceed 200 mg m^{-3} and the alcohol concentration, 1 000 mg m^{-3}. Acetone cells should be fitted with tightly closing lids and fabricated from a non-sparking aluminium-magnesium alloy for reasons of safety. Personnel handling organic solvents must wear protective clothes and gear (such as biological gloves, goggles, and sleeves). In the degreasing area, personnel is not allowed to smoke, to use electric heating appliances, to do work which may cause sparking, or to store oiled clothes and wastes. Spent acetone must be collected in tightly lidded containers for subsequent regeneration or destruction in a designated area. When installing or removing the parts to be joined and jigs, the men doing the job should wear tarpaulin gauntlets.

17.2 Fatigue and Recovery of Personnel

Among the factors causing fatigue to man in industrial environments are the impurities present in the air, noise, vibration, weather conditions, illumination, posture, physical and mental strain, etc. Fatigue can be moderated, health preserved and productivity improved by provision of optimal conditions for work and recreation. As far as health and productivity are concerned, the illumination of the working area is particularly important. Both poor and excessive illumination within the working area and an improper choice of luminaires and their locations can all aggravate fatigue and lead to a higher rejection rate and more frequent injuries. Excessive ambient temperature, too, has a negative effect on man's ability to do work. As has been found, the increase in air temperature from 290-294 K to 308-315 K causes a reduction of 8-19% in labour productivity. Since the mucous membranes and the nervous system in man are highly sensitive to many chemicals present in the industrial atmosphere (acetone, petrol, carbon monoxide, etc.), maximum safe

limits have been established for their concentration. Vibration and noise can sometimes depress labour productivity by as much as 40%. Fatigue can be minimized and recovery promoted by changing posture during the day, doing physical exercises, and reducing noise, vibration, and air pollution on the premises.

17.3 Cleanliness in Vacuum Diffusion Bonding

This refers to measures whose objective is to protect parts to be joined from likely contamination during the production cycle, as the presence of even minute foreign particles on the mating surfaces can markedly impair the quality of bonded joints. This also includes the maintenance of cleanliness in working areas, of clothes, tools and equipment at every step of the bonding operation — from blanking to packing.

CLASSIFICATION OF PREMISES. Under a classification adopted in the Soviet Union the areas that can be used for vacuum diffusion bonding in the manufacture of electron devices are divided into three categories, I, II and III.

Category I refers to hermetically sealed locations isolated from the surroundings with the aid of air locks and a sanitary zone. The climatic conditions in such locations are produced and maintained automatically. Areas in category I are used for especially critical steps in which no dust, organic products, oxides or any other substances detrimental to joint quality may be allowed to reach the mating surfaces. These steps are final cleaning, baking-out and annealing, metal-plating of ceramic components, fit-up, and the bonding proper.

Category II refers to locations used for the closing steps in the preparation of workpieces for final fit-up, such as sealing-off, evacuation, preliminary cleaning and assembly; and diffusion bonding of a wide range of less critical products, repair jobs, heat treatment, preburning (ageing) and testing of electron devices.

Category III refers to locations used for the machining of metal and nonmetal components, preparation of ceramic pastes, application of coatings, painting, etc.

A location in each category is expected to meet specified requirements related to cleanliness and ambient conditions, such as the number of dust particles per litre of air, the number of dust particles settling per square centimetre of a control surface per hour, ambient temperature, relative humidity, and impurity concentration. In Soviet practice, the range of steps and operations that may be carried out in a particular location and the requirements for the latter are determined by the works' chief engineer and product engineer.

In the manufacture of electron devices, the maintenance of specified ambient conditions is essential. The temperature in the working area can markedly affect the quality of the finished product since the components are very small and even minute variations in ambient temperature can bring about marked changes in performance. It is usual to maintain the temperature in work areas at 295 ± 2 K. Air humidity affects many properties of materials and products, especially insulating and dielectric parameters. The relative humidity should be held at 50-65%.

As a way of maintaining proper ambient conditions, locations in Categories I and II should be isolated from one another. No ingress of air from a lower-category

to a higher-category room is permitted. To avoid it, an excess pressure of 24.5-49 Pa should be maintained in the rooms. The air conditioning system should supply air in which the concentration of pollutants does not exceed the specified limits, and the air should be changed at a rate sufficient to maintain the required temperature and to withdraw excess heat generated in the room. Purified air should be admitted to the room in such a manner that the temperature is the same with height to avoid air convection, hot zones, a temperature difference of over 5 K, and draughts or directed air flows which might stir up dust movement.

THE REQUIREMENTS TO BE MET BY PERSONNEL WORKING IN CATEGORY I AND II LOCATIONS. The above requirements with regard to work locations can be met only if the persons working there know and obey relevant rules. Failure to obey the rules at any step in the production cycle might cause rejection of the product at the time of testing or in service.

Persons working in Category I and II locations must keep their clothes, footwear and hands clean (there should be no polish on the nails); leave their clothes, shoes, books and other personal things in the lockers; put on appropriate work clothes; observe personal hygiene; keep clean their work places, tools and instruments; change clothes on leaving in the designated area; at the end of the day tidy up their work places, clean and stow jigs, fixtures, equipment, and tools.

As part of personal hygiene, it is mandatory to wash hands thoroughly. Persons working in Category I locations should wash their hands with water at a temperature of 35°C, using baby soap, rinse them with water at 18-26°C, finally rinse them with distilled water, and dry them on a lint-free clean towel.

In handling the equipment, use should be made of finger caps and gloves which should regularly be washed by boiling in a soda solution and rinsed in deionized water. Clean finger caps and gloves should be stored in bags or boxes.

All medical and sanitary matters should be supervised by a physician who is a specialist in vocational injuries and diseases.

STORAGE OF CLEANED COMPONENTS. Prior to joining, components should be kept in a Category I location. For protection against mechanical damage, dust and other contaminants, cleaned parts should be stored in trays, containers, ampoules, desiccators, and the like, made of nonhygroscopic, noncorroding, lint-free and smooth materials, such as glass, PTFE, PVC, glass-fibre plastics, or metals which do not oxidize in air. It will be a good plan to keep finally cleaned parts in containers of a lighter colour, and uncleaned parts in containers of a darker colour. Finished assemblies and components should be stored in desiccators, clean medical cabinets, cabinets with a controlled atmosphere, or regulated constant-temperature cabinets. Desiccators should be fitted with safety features to avoid ingress of dust from the silica gel or any other sorbent used.

REQUIREMENTS FOR EQUIPMENT, TOOLS AND WORK PLACES. The equipment to be installed in category I and II locations should meet the requirements of vacuum cleanliness. Its compliance with the requirements should be checked and stated in a special report by a commission consisting of managerial representatives and experts in vacuum cleanliness.

Desks and chairs must be made of lint-free materials which do not accumulate static electricity. Equipment should be free from recesses and cracks at joints where

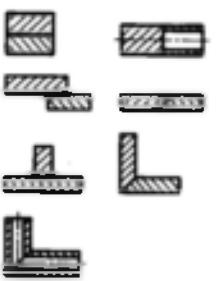
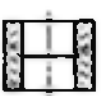
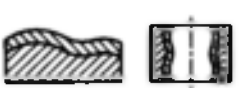
dust might be collected. When the vacuum pumping equipment is located in the same room with the assembly areas, it should be connected to a centralized forevacuum system in order to avoid pollution of the air in the room with oil vapour from forevacuum pumps, and to lower the noise level. If a centralized forevacuum system is not available, facilities should be provided for removal of spent gases from the room.

It is important to keep desks and equipment clean. Personnel should be discouraged from keeping tools, personal things, and food in drawers and desks. All tools and fixtures must be thoroughly cleaned by degreasing, washing, and rubbing with alcohol at regular intervals as prescribed by relevant procedures. Work clothes should be made of a material that would not contaminate the work place and the workpieces with dust, lints, or any other pollutants. The choice of work cloth items depends on the category of the operation and location involved. For those working in category II locations, these may be white or coloured frocks (from nylon, cotton, therylene, or staple fabric given an antistatic treatment), light all leather shoes, cotton caps (for men) or kerchiefs (for women), finger caps (if necessary) or gloves. Work cloths should be taken off on leaving the work area in locker-rooms. Each person should be issued two or three sets of work cloths.

Appendices

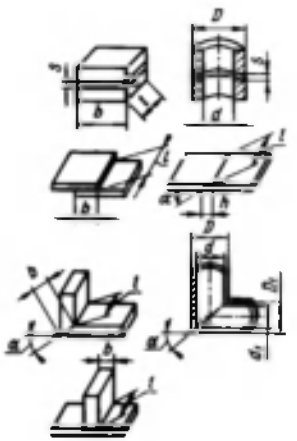
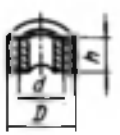
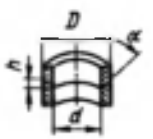
Appendix 1

Types and Symbols of Diffusion-Bonded Joints

Type	Sketch
Flat	
Cylindrical	
Tapering	
Spherical	
Curved	

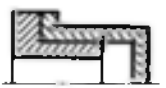
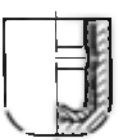
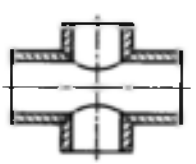
Appendix 2

Dimensions of Diffusion-Bonded Joints

Dimension	Sketch	Joint type
Width, b Height, h Length, l Outside diameter, D Inside diameter, d		Flat
Diameter of sphere, R		Cylindrical
Angle of bevel, α Thickness of interlayer, s		Tapering
Surface area of joint, A		Spherical
Radius of curvature, r		Curved

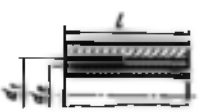
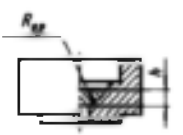
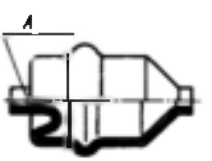
Appendix 3

Combination Bonded Joints

Type	Sketch
Flat-cylindrical	
Flat-curved	
Cylindrical-curved	
Tapering-curved	

Appendix 4

Examples of Bonded Joints

Joint description	Sketch
1. Flat diffusion-bonded joint with interlayers: D = 100 mm; d = 60 mm; S_1 = 0.1 mm; S_{11} = 0.03 mm	
2. Multilayer flat diffusion-bonded joint: l = 160 mm; b = 50 mm	
3. Multilayer cylindrical diffusion-bonded joint: d_1 = 62 mm; d_2 = 50 mm; l = = 100 mm	
4. Spherical diffusion-bonded joint: d_{sp} = = 52 mm; h = 20 mm	
5. Combination diffusion-bonded joint: A = 5125 mm^2	

Appendix 5

Designation of Steels

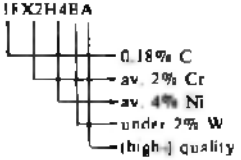
Russian symbol	Element	Chemical symbol
У	Carbon	C
Г	Manganese	Mn
С	Silicon	Si
Х	Chromium	Cr
Н	Nickel	Ni
М	Molybdenum	Mo
В	Tungsten	W
Ф	Vanadium	V
Ю	Aluminium	Al
Т	Titanium	Ti
Д	Copper	Cu
Б	Niobium	Nb
Р	Boron	B
А	Nitrogen	N

NOTES. 1. The first numerals stand for the carbon content in points. The letter that follows stands for the principal alloying element whose content is given in percent. The same order applies to the subsequent letters in the designation.

2. The letter "А" may occur twice: in the middle of a designation it stands for nitrogen, at the end of the designation it stands for "high quality".

Example:

1ГХ2Н4ВА



1. The letter "Э" designates an electrical-sheet steel. The letters "ЭИ" designate an experimental steel.

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